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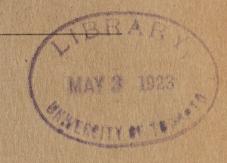
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## TITANIUM

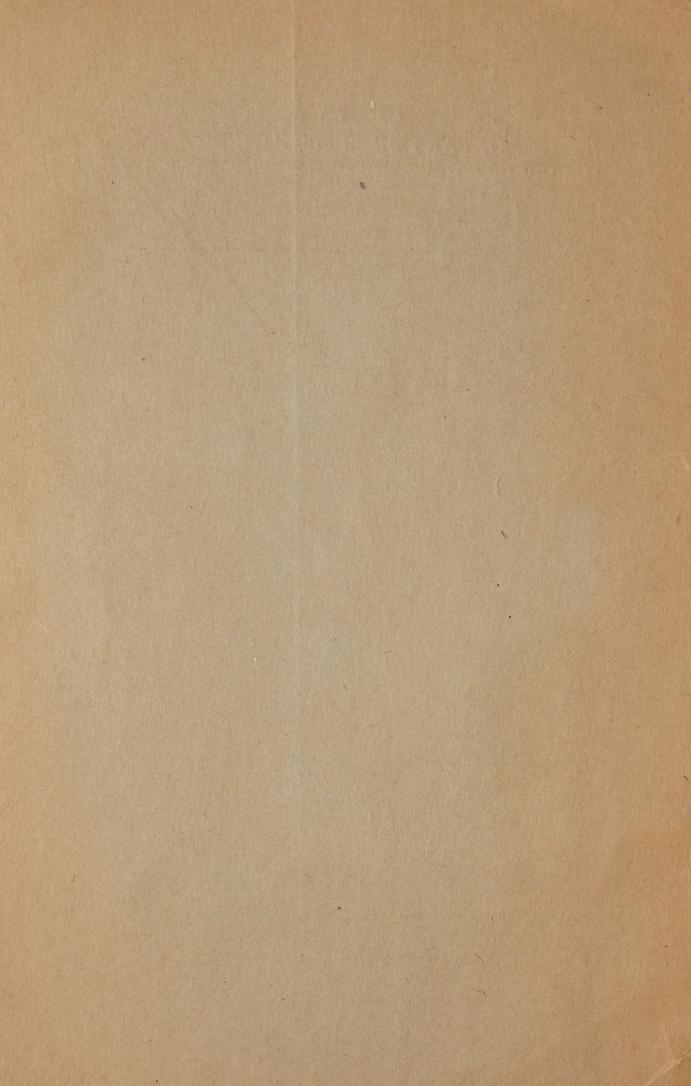


A. H. A. Robinson



OTTAWA F. A. ACLAND PRINTER TO THE KING'S MOST EXCELLENT MAJESTY 1922

No. 579



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## CANADA DEPARTMENT OF MINES

HON. CHARLES STEWART, MINISTER; CHARLES CAMSELL, DEPUTY MINISTER

#### MINES BRANCH

JOHN McLEISH, DIRECTOR

## TITANIUM

BY

A. H. A. Robinson



OTTAWA
F. A. ACLAND
PRINTER TO THE KING'S MOST EXCELLENT MAJESTY
1922

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#### CONTENTS

	PAGE
Introductory	7
PART I	
	4.0
Titanium and its compounds	13 13
History Physical and chemical properties.	15
Preparation of metallic titanium.	17
Occurrence and distribution.	18
Ores of titanium	20
Rutile	21
Ilmenite (titanic iron)	22
Titaniferous magnetite	25 26
Electric furnace smelting of titaniferous magnetites	31
Magnetic concentration of titaniferous magnetites	32
Vanadium in titaniferous magnetites	33
Bibliography	36
PART II	
Titanium occurrences in Canada	41
Rutile occurrences	41
Province of Quebec	41
Charlevoix county Parish of St. Urbain	41
Hull county	44
Templeton township	44
Beauce county	44
Brome county	44
Sutton township	44
Province of Ontario	45 45
Algoma district	45
Province of Nova Sectia	45
Halifax county	45
Kings county	45
Yukon Territory	45
Thistle creek	45 45
Ilmenite occurrences	46
Province of Quebec. Charlevoix county.	46
Parish of St. Urbain	46
Glen pro pect.	47
Bouchard's mine	48
Coulomb's mine	48
The General Electric Co.'s mine	49 51
Furnace mine	52
Lot 641, St. Thomas range	52
Décharge range	53
Quebec Seminary lands	53
Terrebonne county	53
Beresford township	53
Ivry mine	53 56
Mimor occurrences of limenite	56
Province of Quebec.	58
Chicoutimi county	58
Bourget township	58
St. Charles prosp ct	58
Kenogami township	59
$46410-1\frac{1}{2}$ 3	

	PAGE
Lake St. John county	60
Ile d'Alma	60
Saguenay county	60
Bay of Seven Islands	60
River des Rapides	61
Cran de Fer falls; Molson mine	62
Outarde falls	64
Gagnon deposits	64
Marguerite river	65
Clarke City deposits	65
Chaloupe river (Cap Rond)	66
Thunder river	66
St. Maurice county	66
Shawinigan township	66
Grondin, or Shawinigan mine	66
Terrebonne county	68
Beresford township	68
Degrosbois deposits	68
Beauce county	69
St. Charles range	69
Bloc range	70
Hull and Pontiac counties	70
Templeton township	70
Range VI, lot 28	.70
Hull township.	71
Range XI, lot 1	71
Range $X$ , lot $3$	71
Bristol township	71
Range I, lot 22	71
Clarendon township	71
Range VII, lot 27	71
Litchfield township	71
Range V, lot 12	71
Range VIII, lot 10	71
Range X, lots 4 and 5	72
Sheen township, lots 12 and 13	72
rovince of Ontario	72
Leeds county	72
North Crosby township	72
Matthews or Yankee mine	72
South Crosby township	73
Chaffey mine	73
Frontenac county	74
Hinchinbrooke and Bedford townships	74
Eagle Lake or Blessington mine	74
Renfrew county	75
Blithfield township	75
Lot 13, concession I	75
Grattan township Parks property: lot 16, concession VIII	76
Horton township	76
Horton township	76
Concession VI	76
Hastings county	76
Tudor and Lake townships	76
Orton mine	76
Rickett's property Lots 41, 42, 54, and 55, Hastings road	77
Wollaston township	78
Wollaston township  Lots 9 and 10, concession XV.	78
Haliburton county.	78
Glamorgan township.	78
Pine Lake, or Pusey deposit	78
Minden township.	78
Lot 11, concession I.	80
, , , , , , , , , , , , , , , , , , , ,	80

P

	PAGE
Parry Sound district	80
Lount township	80
Lot 132, Rousseau and Nipissing road	80
Lot 144, Rousseau and Nipissing road	80
Lot 136, Rousseau and Nipissing road	80
Nipissing district	80 80
Ottertail brook	80
Sudbury district	80
McNaught township	80
McVittie locations: W.D. 275 and W.D. 276	80
Thunder Bay district	82
Haystack mountain	82
Rainy River district	83
Seine bay and Bad Vermilion lake	83
Mining locations 181 P, 182 P, and 183 P	84
Mining locations, A.L. 25, A.L. 26, A.L. 27, A.L. 28, nd	84
A.L. 29 Mining locations, A.L. 19 to A.L. 25	85
Province of Alberta.	88
Burmis	88
PART III	
Sources, production, and uses of titanium	93
The world's commercial sources and production of titanium minerals	93
Uses of titanium and its compounds	96
Metallic alloys.	97
Ferrocarbontitanium	98
Ferrotitanium (carbon free)	99
Ferrotitanium versus ferrocarbontitanium	100
Titanium in the metallurgy of steel	102
The effect of titanium on cast iron	$\frac{104}{105}$
Cuprotitanium. Ferrosilicotitanium.	106
Ferrocuprotitanium	106
Manganotitanium	107
Incandescent media	107
Arc light electrodes	107
Incandescent lamp filaments	107
Incandescent gas mantles	108
Colouring materials	110
Pigments in the paint industry	110 110
Dyes and mordants in the textile and leather industries	116
Refractory pigments in the ceramic industries	117
Miscellaneous uses	118
Index	121
ILLUSTRATIONS	
ILLUSTRATIONS	
Drawings	
Fig. 1. Sketch map showing the location of the St. Urbain ilmenite deposits	46
2. Sketch map showing location of Ivry ilmenite deposits	55
"3. Sketch map showing location of titaniferous magnetite deposits at Bay of	00
Seven Islands	61
Seven Islands	
iferous magnetite deposits	82
" 5. Diagram of the Carteret-Devaux process	115
Maps	
No. 481. Magnetometric map of the Orton mine and vicinity. Ed. 2 (In poor	1 . (1) =
No. 481. Magnetometric map of the Orton mine and vicinity	ket)
No. 482. Magnetometric map of part of the Seine Bay transferous magnetite deposits	:6
magnetic deposits	

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#### TITANIUM

#### INTRODUCTORY

In the course of the 130 years that have elapsed since the discovery of the element titanium, the attention of chemists and metallurgists has been frequently directed to investigations of both the metal and its compounds; and much has been written concerning their peculiarly useful qualities, and many suggestions made as to their possible applications in the arts and industries. So far, however, the metal has not been utilized to any great extent, and until recent years titanium compounds, as articles of commerce, were almost unknown. Even yet, the amounts so used are comparatively small, and their application

is confined to a few highly specialized lines of industry.

Nevertheless, with the intensified interest now manifest in the possibility of extensive utilization of the so-called rare metals, and with the at least partial removal, through advances in modern industrial chemistry, of some of the economic handicaps under which the preparation of titanium compounds formerly laboured, it is felt by many that titanium, if only on account of its widespread and relatively abundant occurrence, will take a much more important place in industry in the future. In the past, the extraordinary stability of the common titanium minerals, and the consequent almost insuperable difficulties of manipulating them with the appliances then available, militated against the production of useful titanium compounds at a cost low enough to permit of their ordinary industrial use. But the development of the electric furnace and aluminothermic processes for the reduction of refractory oxides has already made ferrotitanium and other titanium alloys commercial products of some importance. Still more recently the researches of Norwegian chemists have led to the commercial development of what promises to be a most valuable titanium pigment. With more attention focussed on the possibilities of titanium and its derivatives, and with the improved appliances and methods daily becoming available to the industrialist, it is not too much to expect a rapid extension of their use.

Though Canada contains, in the province of Quebec, some of the largest deposits of titanium minerals at present known-deposits that will compare favourably as regards size, grade, and accessibility with those of any other country-and though conditions in Quebec would seem eminently favourable to the establishment of a successful titanium industry, such scant notice as has vet been taken of titanium by Canadian scientists and industrialists has been almost entirely confined to those occasions in which its unwelcome presence as a deleterious ingredient of certain iron ores has been forced upon their attention. The ferrotitanium industry, at present the most extensive industry based on the use of titanium, was developed largely in the United States; the manufacture of titanium pigments, which promises to become of still greater importance than the ferrotitanium industry as a consumer of titanium minerals, was developed in Norway, in order to utilize the hitherto practically valueless ilmenite deposits of that country; Canada's titanium industry, if such it can be called, has been limited to the sporadic export of a few thousand tons of ilmenite to the United States.

The available information concerning titanium and its compounds occurs widely scattered through a voluminous literature. By bringing together within small compass a number of the more salient facts concerning them, by outlining the measure of success that has already attended their commercial exploitation, and by drawing attention to the relative importance of the Quebec ilmenite deposits as possible sources of titanium products, it is hoped, through the present bulletin, to arouse an interest that will lead to the investigation by Canadian manufacturers of the possibilities of the titanium industry in Canada. The principal Canadian deposits lie within one hundred miles of the industrial centres of Montreal and Quebec, in districts where hydro-electric power is abundant and cheap, and where there is easy access to wide markets. Up to the present, the manufacture of ferrotitanium has been, commercially, the most successful titanium industry, but possibly market conditions are not particularly favourable to its establishment in Canada. The same objection, however, would not appear to hold in the case of titanium pigments. The commercial position of the latter, it is true, cannot yet be said to be fully assured, but the opinion of persons well qualified to form an accurate estimate of the future importance of titanium white in the commercial world can be gathered from the following extracts from the annual report of the National Lead Company, of New York, for the financial year ending December 31, 1920:—

The significance of the above extract lies in the fact that the National Lead Company is one of the largest producers of lead pigments in the United States and is one of four companies that among them control 95 per cent of the white lead output of that country;<sup>2</sup> and in the further fact that white lead is the material with which titanium white will come into most active competition.

In connection with the commercial outlook for titanium white, the possibility of legislative action with a view to the prohibition of the use of its rival, white lead, in paints, is also worth noting. The following information in regard to this aspect of the matter has been furnished by Mr. Gerald H. Brown, Assistant Deputy Minister of Labour:—

A draft convention, looking to the prohibition of the use of white lead in interior house painting, and to the regulation of its use in exterior painting, was adopted at the League of Nations' International Labour Conference in Geneva in the month of November last (1921). This draft convention, as well as other proposals of the International Labour Conference, will be submitted in due course to the Government of Canada for the enactment of legislation for acceptance or otherwise. As you are aware, the Government of the United States is not represented in the League of Nations, and

<sup>&</sup>lt;sup>1</sup> Before the war, in addition to two plants in the United States, fifteen manufacturers in Germany, three in Great Britain, two in France, and several others in Switzerland and Sweden were producing titanium alloys.

<sup>&</sup>lt;sup>2</sup> Sales of white lead by the National Lead Co. amounted to over \$21,000,000 in 11 months, in 1990.

that country did not, therefore, participate in the recent conference at Geneva. The Government of Great Britain, however, was represented, and its representative voted in favour of the proposed prohibition and regulation of the use of white lead in painting.

I may add that stringent laws relative to the use of white lead in painting have been

adopted in France, Austria, and Greece, among the European countries.

Titanium white, unlike white lead, is extremely inert and absolutely nonpoisonous. There is no obvious reason why it could not be produced in Canada just as well as in Norway or the United States. The quantity of raw material consumed by such an industry might not be large and perhaps would not go far to swell the total of our mineral output, but the importance of a substance to the community should not be gauged by the amount consumed but by the results which arise from its application in the arts. The matter of a few thousand tons of ilmenite mined annually for export is a thing of little moment in comparison with the advantages to be derived from working up the same amount into finished products. The establishment of one titanium industry in the country would doubtless lead to the development of others, and to an expanding use of an at present idle mineral resource. Looked at from any point of view, the question of the establishment of a titanium industry in Canada is well worthy the serious consideration of Canadian manufacturers and technicians. The matter has already been taken up in France, by French chemists, with a view to the utilization of ilmenite deposits in Madagascar.

The bulletin that follows is of necessity almost entirely a compilation. Such original material as has been added has been used to supplement or modify older descriptions of Canadian titanium occurrences, most of which have been visited and examined by the writer. It is believed that the references throughout the text are sufficiently numerous and varied to cover the whole field, and serve as a guide to any who may wish to go further into any particular

phase of the subject.



# PART I TITANIUM AND ITS COMPOUNDS



#### TITANIUM AND ITS COMPOUNDS

(N.B.—To avoid a needless repetition of footnotes, references to authorities in Part I of this report are made by means of figures, corresponding with those of the numbered list on pages 36-38 at the end of Part I.)

#### HISTORY 1, 2, 3, 4.

The existence of the element we now know as titanium was discovered at the beginning of the last decade of the eighteenth century, by the Rev. Wm. Gregor (or McGregor), while he was investigating a peculiar mineral found in the form of a black sand, at Menachan, in Cornwall. The black sand he named menachanite (menaccanite) after the locality in which it was found, and the new element he called menachite. Four or five years later, in 1794 or 1795, the German chemist Klaproth, while investigating the composition of the mineral rutile, discovered a new metal, to which, on account of the strength of the chemical combination in which it was held, he gave the name titanium—in allusion to the Titans of ancient Greek mythology, the incarnation of natural strength. In a subsequent investigation of ilmenite, in 1797, Klaproth recognized that titanium was identical with the menachite of Gregor. Titanium, however, is the name that has been universally adopted.

Attempts to isolate the new element were first made by Lampadius, in 1797; later by Berzelius, and a number of other investigators. Due to its high fusion point and strong affinities for nitrogen and carbon as well as for oxygen, these earlier attempts to reduce titanium from its compounds were unsuccessful and usually resulted in the production of some of the nitrides or carbides, which, on account of their metallic lustre, were commonly mistaken The copper-red compound that we now know to be a for the metal itself. cyanonitride of titanium was, until its true composition was demonstrated by Wöhler, in 1849, commonly believed to be metallic titanium. Prior to the work of Hunter,8 the results of which were published in 1910, it seems very doubtful whether pure metallic titanium had ever been prepared. Up to that time the nearest approach to the pure element was probably that obtained by Moissan, by fusing carbon with an excess of titanium dioxide in the electric furnace at very high temperatures, an account of which was published by him in 1895. Moissan's final product was free from nitrogen and silicon, but contained two per cent carbon, probably as the carbide, TiC, and also some oxygen: it was, therefore, far from being pure titanium. Titanium produced by methods other than Moissan's was contaminated to a still greater extent, usually by the monoxide, TiO, and by nitrogen.

Hunter<sup>8</sup> attempted to prepare pure titanium by a number of the methods previously employed, among others that of Moissan, but was unsuccessful until he adopted one, earlier used by Nilson and Peterson, <sup>10</sup> which consists essentially in reducing titanium tetrachloride (TiCl<sub>4</sub>) by means of sodium in an air-tight steel cylinder. The best result that Nilson and Peterson had been able to obtain was a product containing only about 79 per cent titanium in the form of pure metal; the chief impurity being oxygen, present presumably as titanium monoxide (TiO). Hunter, however, by exercising rigorous care to ensure the exclusion of air from the apparatus used, succeeded in obtaining in this way metallic titanium practically 100 per cent pure.

The Nilson-Peterson method as carried out by Hunter is the standard laboratory method for the production of pure metallic titanium but is much too costly for its production for ordinary industrial purposes. Titanium alloyed with other metals is produced commercially either by Rossi's 11 electric furnace process or by the Goldschmidt 2 alumino-thermic method. Its manufacture in a substantially pure state by a process cheap enough to be commercially satisfactory has apparently not yet been accomplished, and no

industrial use for the pure metal has yet been found.

Though the possibility of utilizing various compounds of titanium in the arts began to be suggested soon after the discovery of the element, it is only within comparatively recent years that any noteworthy progress has been made in this direction. For example, in 1846 the use of ferrocyanide of titanium as a green paint was proposed as a substitute for the poisonous arsenical copper green then so commonly used; in 1861 specifications for a patent covering the use of certain compounds of titanium as pigments, or colouring materials, were deposited in London by F. Versamm; about this same time, 1861, Mushet took out several patents for certain methods of utilizing the ores of iron and titanium and for obtaining alloys of the two metals, thus foreshadowing the present extensive use of titanium alloys in the treatment of iron and steel. 13

Between March 1859 and December 1861 thirteen English patents were granted to Robert Mushet for alleged improvements in the manufacture of iron and steel, in all of which titanium plays the chief part. The eleventh patent (No. 2637, Oct. 22, 1861) covers the manufacture of a triple metallic compound, or alloy, of iron, titanium, and manganese, containing also carbon; in the twelfth (No. 2744, Nov. 1, 1861) he is granted the exclusive right of alloying cast steel or "homogeneous iron" with the triple alloy specified in the previous patent; the essence of the thirteenth, and last, (No. 3116, Dec. 12, 1861) consists in the addition of "titanic pig-metal" to ordinary pig iron, cast iron or refined iron, or any mixture of these in a puddling furnace with a view to improving the quality of the malleable iron, bar iron, or puddled steel which may be produced from such mixtures. To what extent Mushet anticipated the subsequent work of Rossi it is impossible to say, but the procedure he outlines is sufficiently similar to modern practice in the use of ferrotitanium to call for remark. What is certain is that Mushet, despite most vigorous efforts, failed to convince the ironmasters and metallurgists of his day of the surpassing excellence of his titanic steel, and the matter was dropped.87

Specially desirable qualities in the irons made from titaniferous ores—though the modern tendency is to attribute these qualities to factors other than the presence of titanium—as well as certain undesirable conditions produced by titaniferous ores in the blast furnace, early attracted attention to titanium in the metallurgy of iron. But, until Rossi's<sup>14</sup> work in the eighteen-nineties had led to the production of ferrotitanium and demonstrated its value for the purification of steel, the industrial use of titanium compounds was almost negligible and confined chiefly to the ceramic industry, in which small quantities

were employed as refractory colouring material.

The credit for the establishment of the ferrotitanium alloy industry, by far the most important titanium industry in existence to-day, is largely due to Auguste J. Rossi, a French metallurgist resident in the United States. Rossi's first essays in connection with titanium were made in an attempt to demonstrate

the feasibility of using titaniferous ores for the production of iron in the modern blast furnace<sup>15, 16, 17</sup>; an attempt in which he achieved technical success, but which for various reasons was not productive of commercial developments. His work on the smelting of titaniferous ores led him next to try to produce titanium-iron alloys by reducing iron and titanium together in the blast furnace, but he found himself unable to make an alloy high in titanium in this way. He then turned his attention to the electric furnace, and at the high temperatures obtainable in it succeeded in evolving an economical process for the manufacture of the high titanium alloys, ferrotitanium and ferrocarbontitanium. The company formed to exploit these and other titanium products is the Titanium Alloy Manufacturing Company of Niagara Falls, New York.

Rossi appears to have produced ferrotitanium by an industrially practicable process prior to 1895, but the possibilities of the new alloy were slow in obtaining recognition. What may be regarded as its first extended application began at the Maryland Steel Works, in 1907, 18 for the purification of steel, and it was used for the treatment of cast iron at the car wheel foundry of the Norfolk and Western railway at Roanoke, Virginia, in 1908. 19 Since then its use in the treatment of steel and iron has increased with great rapidity, and its manufacture has become a large and important industry. It is estimated that in 1918 some 2,400,000 tons of steel of all kinds were treated with ferrotitanium in the United States. 20

## PHYSICAL AND CHEMICAL PROPERTIES OF TITANIUM AND ITS COMPOUNDS 1, 2, 3, 4, 8, 21.

Titanium belongs to the same chemical group of elements as silicon, zirconium, cerium, and thorium. It resembles these in being tetravalent, with a lower valence in some cases; like zirconium it may behave either as a weak base-forming or weak acid-forming element. In its behaviour towards reagents it somewhat resembles niobium and tantalum. In a general way its chemical properties are similar to those of silicon, but it differs from that element in that it is more active in its combining qualities, since it unites readily with a greater number of elements and forms a greater number of different compounds with each. It is also in some respects more energetic in its action than silicon, uniting with oxygen, nitrogen, chlorine, and bromine with incandescence.

As prepared by Hunter,<sup>8</sup> metallic titanium does not differ in outward appearance from polished steel. It is hard and brittle when cold and breaks with a steely fracture; but it is malleable at a low red heat and at that temperature may be readily forged like red hot iron. Small beads of the metal may be flattened out without disruption even when cold. Attempts to draw it into wire through a heated draw plate were unsuccessful.

Its atomic weight is 4.81, and the specific gravity, as determined by Hunter, 4.5.8 Hunter also determined the melting point as being between 1800° C. and 1850° C. Moissan<sup>9</sup> and Rossi<sup>22</sup> both give much higher figures, the latter placing it between 2700° C. and 3000° C., a discrepancy probably explained by the fact that Moissan and Rossi worked with alloys of the metal with its compounds, not with pure metal.

Moissan<sup>23</sup> states that titanium combines with oxygen with incandescence at a temperature of 610° C., and Hunter<sup>8</sup> found that a rod of the metal burned

in air at a temperature of 1200° C. when an electric current of 50 amperes was sent through it, the heat of oxidation being sufficient to melt the oxides formed. Cahen and Wootton<sup>24</sup> say that when heated in air it burns with extreme

brilliance, hence has been used in pyrotechny.

According to Browning,<sup>3</sup> titanium does not decompose water at ordinary temperatures and acts but slightly on heated water. It is soluble in cold dilute sulphuric acid, in aqua regia, in hot concentrated hydrochloric acid, and, more slowly, in nitric acid; hydrofluoric and acetic acids also attack it. On heating, it combines with the halogens and oxygen with incandescence, to form halogen salts and oxides, and with sulphur and carbon to form sulphides and carbide. It combines with nitrogen with avidity; and when heated to 800° C. in an atmosphere of that gas titanium, especially in the powdered form, burns with incandescence to nitrates. Roberts-Austen<sup>25</sup> states that this is the only known instance of vivid combustion in nitrogen.

Titanium has the power of forming numerous compounds having a variety of colours of peculiar richness, such as red, green, yellow, and blue. The sulphide prepared by the action of sulphuretted hydrogen on the bi-chloride is a beautiful bronze substance that crystallizes in scales with a metallic lustre and resembles mosaic gold. The different nitrides studied by Wöhler<sup>26</sup> were violet, golden-yellow, and copper-coloured; and, according to Karsten,<sup>27</sup> titanic acid fused with metallic tin in a vitrifiable base gives rise to a beautiful deep blue enamel, thus resembling smalt prepared with the oxide of cobalt.

The compounds of titanium also vary widely in their state of aggregationfrom the extraordinarily infusible cyanonitride, to the fluid chloride, and the volatile hydrofluoride.<sup>28</sup> Typical compounds are, according to Browning<sup>3</sup>:—

Oxides: TiO, Ti<sub>3</sub>O<sub>4</sub>, Ti<sub>2</sub>O<sub>3</sub>, Ti<sub>7</sub>O<sub>12</sub>, TiO<sub>2</sub>, Ti<sub>2</sub>O<sub>5</sub>, TiO<sub>3</sub>. Hydroxides: Ti (OH)<sub>4</sub>, Ti (OH)<sub>6</sub>.

Chlorides: TiCl<sub>3</sub>, TiCl<sub>4</sub>.

Bromide: TiBr4. Iodide: TiI4. Fluoride: TiF4.

Titanofluorides: R2TiF6, etc. Sulphides: Ti2S5, TiS2.

Sulphates: Ti<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, Ti(SO<sub>4</sub>)<sub>2</sub>. Nitrides: Ti<sub>3</sub>N<sub>4</sub>, Ti<sub>5</sub>N<sub>6</sub>, TiN<sub>2</sub>.

Carbide: TiC. Silicide: TiS.

Titanates: RTiO<sub>3</sub>, R<sub>2</sub>TiO<sub>3</sub>.

Acids: (see Hydroxides) H<sub>2</sub>TiO<sub>3</sub>.

It shows only slight affinity for phosphorus and arsenic.

As for the oxides of titanium, the most important are: the dioxide, TiO2; the sesquioxide, Ti<sub>2</sub>O<sub>3</sub>; and the peroxide, TiO<sub>3</sub>. The monoxide, TiO, also, has been prepared, but the existence of some of the others is doubtful.2

Titanium dioxide is trimorphous and occurs in nature as three different minerals; rutile, brookite, and anastase. It fuses at the temperature of the oxy-hydrogen flame to a thin liquid that solidifies on cooling to a confused crystalline mass.2 It behaves as an acid flux, uniting with iron oxide, aluminum, calcium, and other metals to form titanates, which appear to lower the melting point of the silicates of these metals.<sup>30</sup> Its heat of formation is given by Mixter as about 218,000 and in point of stability no other known compound surpasses it.<sup>31</sup>

Titanium oxide, like the metal, when heated in the presence of nitrogen, is transformed into titanium nitride<sup>8, 9, 26</sup>; and titanium cyanide if carbon also is present<sup>32, 7, 33</sup>. It is probably in this way that the various mixtures of nitride and cyanide, somewhat vaguely termed cyanonitride of titanium, are produced when iron ores containing titanium are smelted in the blast furnace. Under these conditions the cyanonitride forms as small, brilliant, copperationed cubes found lining cavities in the slag and pig iron and replacing mortar in the firebrick furnace lining. The crystals are hard enough to scratch glass and are highly refractory. Masses up to 80 pounds in weight of this material have been found in the blast furnace.<sup>2</sup> It was noted in 1822, by Wollaston,<sup>34</sup> who in common with other early investigators took it for metallic formium. Wöhler,<sup>7</sup> in 1849, showed that it contained both nitrogen and cyanogen, and ascribed it to the formula Ti(CN)<sub>2</sub>·3Ti<sub>3</sub>N<sub>2</sub>. It is highly resistant to teids, being attacked only by a mixture of hydrofluoric and nitric acids. Its specific gravity is 5·28.<sup>2</sup>

Titanium forms only one stable carbide, TiC. Titanium carbide when heated to redness, either in oxygen or in the air reacts with oxygen in a manner not unlike the metal.<sup>9</sup> It is harder than carborundum. This compound has been recognized in pig iron and in ferromanganese, but is said to be present only in minute quantity in ferrotitanium, that is, in pig iron carrying more than about 10 per cent titanium.<sup>35</sup> Analyses of ferrocarbontitanium published by the Titanium Alloy Manufacturing Company show their product to contain graphite up to 9.6 per cent, with less than 0.15 per cent combined carbon.<sup>29</sup> In fact the action of titanium is said to be to precipitate the carbon from iron and steel, acting in this way much as does silicon.<sup>35</sup>

#### PREPARATION OF METALLIC TITANIUM<sup>8</sup>, 2, 29, 36.

Due to its high fusion point and strong affinities for nitrogen, carbon, and oxygen, the production of the metal, titanium, is beset with difficulties. The earlier attempts to isolate it resulted for the most part in the production of various nitrides, which from their metallic appearance were mistaken for the metal; thus, Wollaston mistook the cyanonitride in 1823.<sup>34</sup> Berzelius,<sup>6</sup> by reducing potassium titanifluoride with potassium obtained a black powder that on polishing showed a metallic streak, which he took for titanium; this substance, however, was insoluble in hydrofluoric acid, and so, whatever else it may have been, it was not titanium, for that metal is immediately soluble in hydrofluoric acid. Rose<sup>37</sup> produced a dark blue or black powder by heating lone, or by reducing with metallic sodium the compound TiCl4.4NH3; the product he obtained we now know to be Ti<sub>3</sub>N<sub>4</sub>. Wöhler,<sup>7</sup> in 1849, and Wöhler and Deville,<sup>26</sup> in 1857, in their attempts to produce elementary titanium by the reduction of titanifluorides by the alkali metals, succeeded only in obtaining the nitrides. Up to this time no analyses of the materials produced by the experimenters appear to have been made, and confusion of the pure metal with its nitrides appears to have been general.

Nilson and Peterson<sup>10</sup> (1887) tried to prepare metallic titanium by the reduction of TiCl<sub>4</sub> by means of sodium in an air-tight cylinder of steel. What 46410-2

they obtained was in the form of yellow scales containing 94.73 per cent titanium, the chief impurity being oxygen. As this was present presumably combined with titanium as titanium monoxide, TiO, the material produced would contain only 78.92 per cent of the metal as such.

Moissan (1895)<sup>9</sup> by reducing TiO<sub>2</sub> at the extreme temperature of his electric furnace produced a material containing, in addition to titanium, 5 per cent of carbon. Re-heating this with TiO<sub>2</sub>, the carbon was reduced to 2 per cent, and the final product was free from nitrogen. This appears to be the

purest metallic titanium that had been obtained up to that time.

Hunter<sup>8</sup> (1910) repeated the experiments of Berzelius, Wöhler, and Deville, but was unable to obtain a product containing more than about 73 per cent of titanium by any of the methods used by them. He next tried the reduction of titanium dioxide by carbon, but succeeded only in obtaining a bluish-purple material carrying varying amounts of TiC and TiO, and concluded that the possibility of producing pure titanium by this method was doubtful. Finally, he repeated the experiments of Nilson and Peterson, and by taking extraordinary precautions to exclude air from the apparatus, succeeded in getting

practically pure metallic titanium.

In the Nilson-Peterson method as carried out by Hunter, the tetrachloride of titanium used was made by chlorinating titanium carbide, the chloride formed being re-fluxed in a current of nitrogen to remove chlorine and then repeatedly distilled with the rejection of end fractions. The fraction boiling between 136°C. and 137°C. was then shaken repeatedly with sodium amalgam and mercury and re-distilled, the product being a water-like, colourless substance boiling at a constant temperature. Five hundred grammes of this titanium tetrachloride and 245 grammes of sodium, theoretically necessary for its reduction, were mixed and placed in a steel bomb capable of withstanding a total internal pressure of 80,000 pounds. The charged bomb was brought to a low red heat, when an almost instantaneous reaction took place. After leaching the contents of the cooled bomb with water, titanium alone remained, partly in the form of a fine grey powder, partly as small rounded metallic beads. The heat of the reaction had been sufficient to fuse most of the metal into globules. Two analyses of the metallic beads yielded respectively 99.9 per cent and 100.2 per cent titanium.

The method used by Hunter is still standard for the production of pure metallic titanium in the laboratory, but, like many laboratory methods, is much too costly for the production of titanium for ordinary industrial use. For this purpose some process comparable in cost with Goldschmidt's thermit process, or Rossi's process for the production of ferrotitanium, is necessary. Impure titanium can be produced by both these processes—to be described later in connection with ferrotitanium alloys. Huppertz, <sup>38</sup> Borchers, <sup>39</sup> Lodyguine, <sup>40</sup> and Gin, <sup>41</sup> have also developed processes that appear to have commercial possibilities; but a satisfactory process for the manufacture of substantially pure titanium in a commercial way has not yet been put in operation. <sup>1</sup>

#### OCCURRENCE AND DISTRIBUTION OF TITANIUM

It has been customary to class titanium with the rare elements, but this description of it is true only in the narrow sense that it is seldom met with in nature in easily recognized forms or concentrated in such quantity in any

one locality as to attract attention. In every other respect it is far from rare. Clarke43 estimates that it constitutes 0.44 per cent of the solid crust of the earth, and of the eighty-odd elements comprised therein it ranks ninth in relative abundance. It is more abundant than the common elements carbon, phosphorus, sulphur, and manganese, and much more so than the common useful metals lead, copper, and zinc. In fact the only metals in common use that surpass it in this respect are aluminum, iron, and magnesium. It is also one of the most widely distributed. Nevertheless, a much smaller proportion of the aggregate bulk of the titanium contained in the earth's crust is found concentrated in separate deposits than is the case with the common useful metals. The greater portion of it occurs diffused through such a vast mass of rock as to make its recovery economically hopeless, so that, leaving out of consideration its characteristic refractoriness, its abundance in nature is not altogether a measure of its ultimate availability for the uses of man. Available supplies, however, are far in excess of any present or prospective demands likely to be made on them

Titanium occurs in nature in a great variety of mineralogical forms. Watson and Taber, in their "Report on the Geology of the Titanium and Apatite Deposits of Virginia" 136, list over sixty minerals of which titanium is one constituent. Most of them, however, are of merely academic interest.

It occurs chiefly as oxides, titanates, and silico-titanates; and enters also into the composition of many silicates and some niobates and tantalates. It is never found native. As the oxide, rutile, it is found in some rock magmas rich in silicon and relatively poor in basic elements; in most rock magmas where there is sufficient calcium and titanium to form calcium titanite, it appears as perovskite (CaTiO<sub>3</sub>); in magmas lowest in silicon and richest in iron it forms ilmenite, or iron titanite (FeTiO<sub>3</sub>); in the silicates, it either replaces silicon in the acid radicle or enters as a weak base element.<sup>21</sup> It is of general, perhaps invariable, occurrence in magnetic iron ores of directly igneous origin, either in the form of ilmenite intimately intergrown with the magnetite, or replacing part of the iron in the magnetite molecule itself (titano-magnetite). It is seldom found in any considerable quantity in nematite.<sup>1</sup>

As minor accessory rock constituents, often in only microscopic quantity, titanium minerals are found in all classes of rock—igneous, metamorphic, and sedimentary. In igneous rocks, and in the sediments derived from them, it is almost universally present; it is a commoner element in these than any other element of the titanium group except silicon. Of 800 igneous rocks analysed in the laboratory of the United States Geological Survey, 784 contained titanium.<sup>43</sup> In this connection, it occurs in largest amount in rocks lowest in silicon and richest in iron, and its affinities in the magma seem to be rather with iron than with magnesium, and still less with calcium. Igneous rocks high in calcium and potassium seldom contain much titanium.<sup>44</sup> It has been stated that iron ores when associated with titanium are comparatively free from phosphorus,<sup>45</sup> but there are many exceptions to this rule, and it would be unwise to conclude, merely because an ore is titaniferous, that it is therefore low in phosphorus.

Titanium minerals are also of common occurrence in gneisses, mica schists, phyllites, shales, sandstones, limestones, and dolomite; and in sands composed of heavy resistant minerals, such as gold-bearing sands and gravels, monazite sands,

and black magnetic sands<sup>43, 46, 47</sup>. It is of widespread occurrence in clays,<sup>48</sup> the fusibility of which is said to be increased and the refractory properties injured thereby. As much as two per cent titanic acid has been found in some fireclays,<sup>49</sup> and as much as nine per cent in some bauxitic clays from Georgia.<sup>50</sup> It is present in most fertile soils and has been detected in certain varieties of coal, in mineral waters, in meteorites, in certain woods, in the ashes of plants, in cow-peas, in cotton-seed meal, in human blood, in the bones of men and animals, and, by means of the spectroscope, in the atmosphere of the sun.<sup>51</sup>

All the known deposits of titanium minerals that are of any commercial importance, however, are found either in or closely associated with basic eruptive rocks of the gabbro family, and, generally speaking, occurrences of titanium are decidedly more extensively distributed in basic than in acid rocks.

#### ORES OF TITANIUM

Of all the numerous titanium-bearing minerals only three, or at most five, are known to occur under conditions that entitle them to be classed as possible ores of titanium. These are: rutile, ilmenite, titaniferous magnetite, and possibly titanite, and perovskite. The actual commercial ores are rutile and ilmenite. Titaniferous magnetite may be regarded as low-grade ilmenite and a potential ore; titanite and perovskite are sometimes listed among the titanium ores,<sup>24</sup> but it is doubtful if they ever occur in deposits large enough to be of economic importance.

Rutile and ilmenite are formed mainly under conditions involving high temperatures; either during the original solidification of igneous rocks or as constituents of the pegmatites which follow the crystallization of the main igneous masses. Titaniferous magnetites, which are formed in many basic igneous rocks by the segregation of certain iron-bearing materials, contain in the aggregate large quantities of ilmenite not commercially available under present conditions.

The rutile of commerce, carrying up to 59 per cent of titanium, is the purest and most generally desirable of the ores of that metal; it is also the least plentiful and most costly. Ilmenite, carrying up to 30 or 35 per cent titanium, with about twice as much iron, is cheaper and much more plentiful than rutile, and is the material commonly used in the manufacture of ferrotitanium alloys, where the presence of iron is not objectionable; but for purposes in which the presence of much iron is a detriment rutile is used. Titaniferous magnetite seldom carries enough titanium to be used with advantage, even for the production of ferrotitanium, in competition with the richer ilmenite.

For industrial purposes the only distinction between ilmenite and titaniferous magnetite is in the titanium content. Ore classed as ilmenite generally carries 18 to 24 per cent and upwards of titanium, with from 1.6 to 2.6 times as much iron, while titaniferous magnetite seldom carries more than 15 per cent titanium, and the ratio of iron to titanium usually runs from 4.0 to 6.0. Magnetite carrying less than 3 per cent titanic acid (TiO<sub>2</sub>), an amount that does not interfere with its use in the blast furnace, is usually classed with the ordinary iron ores.<sup>52</sup>

The relative commercial values of rutile and ilmenite as reflected in current market quotations (see Engineering and Mining Journal, Nov. 5, 1921), show that at the present time titanium combined as rutile is worth over five times as

much as that occurring in ilmenite, the quoted prices being 1½ to 2 cents per pound for ilmenite containing 52% TiO<sub>2</sub>, and 12 cents per pound for rutile carrying 95% TiO<sub>2</sub>. This works out to four cents per unit titanium in the case of ilmenite, and about twenty-one cents per unit titanium for rutile. The difference in value is to be ascribed in part to the greater rarity and greater cost of production of rutile, and in part to its comparative freedom from iron and consequent suitability for purposes for which ilmenite is not adapted.

#### RUTILE

Composition Titanium dioxide, TiO2.

Hardness 6 to 6.5

Lustre Metallic to adamantine.

Cleavage Distinct.

Fracture Sub-conchoidal to uneven: brittle.

Streak Pale brown.

Colour Reddish-brown to red (by transmitted light the mineral

appears deep red); sometimes yellowish, bluish, violet,

black, and green.

It is transparent to opaque.

Varieties: ilmenorutile, dicksbergite, strüverite, nigrine, and sagonite.

The specific gravity of the ordinary red variety is 4.18 to 4.25; in one black variety it is 5.2.

Rutile crystallizes in the tetragonal system, commonly as short stout prisms, or elongated prisms frequently showing striated prism faces. It also occurs in acicular, hair-like forms, and in grains and masses of variable size.

It is infusible before the blowpipe and insoluble in acids, but is decomposed by alkalis and alkali carbonates.

The percentage composition of theoretically pure rutile is: titanium, 60 per cent, oxygen, 40 per cent. Actually it usually contains from 54 to 59 per cent titanium (equivalent to 90 to 99 per cent TiO<sub>2</sub>), minute quantities of vanadium, and iron. This last, in some black varieties, may be present to the extent of 10 per cent. More rarely it carries also a little chromium and tin.<sup>36</sup>

Rutile occurs in igneous, in metamorphic, and in sedimentary rocks; in the first both as a pyrogenetic constituent and as an alteration product. It is common in such metamorphic rocks as gneiss, mica-schist, amphibolite, and phyllite; in some sedimentaries;<sup>47</sup> in some rocks produced by contact metamorphism; and in veins, both metalliferous and non-metalliferous.<sup>53</sup> Lindgren classifies it as one of the persistent minerals, that is, one formed under all conditions from those obtaining at great depth to those near the surface: igneous conditions; pegmatitic conditions; contact metamorphic conditions; deeper, middle, and upper vein zone conditions.<sup>54, 55, 56</sup>

It is ordinarily a very stable mineral, but, according to Van Hise, under certain circumstances it alters to hematite, ilmenite, and titanite; and Watson and Taber note the alteration of rutile to a substance resembling leucoxene.<sup>36</sup>

The mineral associates of rutile include a wide range of species. As a product of crystallization in rock magmas it is associated with the common rock-forming silicates and quartz; in metamorphic rocks it is usually accompanied by such heavy metamorphic minerals as cyanite, magnetite, and ilmenite; in veins the commonest associate of rutile is probably quartz together with ilmenite

and other titanium minerals and oxides of iron. In some veins and pegmatites the association includes minerals usually regarded as having been formed by pneumatolysis, for example, the rutile found in the apatite veins of Norway and Sweden. In alluvial and water-sorted sands, rutile is accompanied by such heavy resistant minerals as ilmenite, magnetite, chromite, monazite, garnet,

zircon, and spinel.36

Widespread in its occurrence and general in its associations rutile shows little tendency to form separate deposits. At less than half a dozen widely separated localities in the world is it known to occur in sufficient quantity to be of actual or prospective commercial value. The localities in which economically important deposits have been found are: Kragerö, in southern Norway; Nelson county, Virginia; St. Urbain, in Quebec, Canada; and Mount Crawford in South Australia. At the first three places, the rutile deposits are genetically connected with gabbro or anorthosite; the Mount Crawford occurrence, judging from the meagre description available, appears to be in a pegmatite dike. Commercially promising deposits have also been found in the weathered outcrops of pegmatite dikes in Goochland and Hanover counties, Virginia.<sup>36</sup>

Until a few years ago the Kragerö deposits supplied practically all the world's rutile. At present the more recently discovered Virginia deposits are the world's greatest producers. These are apparently of greater extent and are said to be capable of yielding a higher grade product than those at

Kragerö.<sup>36</sup>

The St. Urbain deposit, though promising, has been worked to only a very slight extent in a tentative way and is practically undeveloped. The rutile is there irregularly distributed through great masses of ilmenite, or titanic iron, so that considerable exploration will have to be done before an opinion can be formed as to whether or not it is capable of constituting a dependable source of supply.

A little rutile has been shipped from the Australian occurrence near Mount Crawford,<sup>58</sup> but the available description of it gives no very definite informa-

tion as to its possibilities.

In the past, in addition to that produced in Norway, small quantities of rutile were recovered as a by-product of washing monazite sands in the Carolinas, (U.S.A.), and in Brazil, and a little is now being obtained in that way in Florida. Crystals of rutile, for museum purposes and for sale to dealers in mineral specimens, were formerly mined in a small way in Pennsylvania, Georgia, and the Carolinas, and, in Pennsylvania, a few hundred pounds annually were picked out of the detritus of rutile-bearing rocks to be used for tinting artificial teeth. Crystals suitable for gems<sup>57</sup> have also been obtained in North Carolina, Georgia, and others of the United States.

In Europe, outside of Norway, the best known localities for rutile are Carinthia and Transylvania.<sup>24</sup>

#### ILMENITE. (TITANIC IRON)

Composition Variable. The formula usually given is FeTiO3.

Hardness 5 to 6. Cleavage Undeveloped.

Fracture Conchoidal, brittle.

Lustre Sub-metallic to metallic.
Streak Black to brownish-red.

Colour Iron black.
Opaque.

May be slightly magnetic, but is seldom appreciably so by ordinary tests. It becomes magnetic on heating.

Ilmenite crystallizes in the hexagonal system, as rhombohedra, but is rarely seen in good megascopic crystals. Commonly it occurs in embedded grains and masses, sometimes showing a platy structure with irregular to hexagonal outlines. It is also found loose, as sand.

It is fusible only with difficulty before the blowpipe. It is difficultly soluble in acids but is decomposed by fusion with bi-sulphate of potash. If it is fused with sodium carbonate and the melt dissolved in hydrochloric acid, the solution, boiled with tin, becomes violet.

The chemical composition of ilmenite is decidedly variable. It is customary to ascribe to the theoretically normal mineral the formula FeTiO<sub>3</sub>, corresponding to TiO<sub>2</sub>, 52·7 per cent, and FeO, 47·3 per cent; many analyses correspond more closely to a general formula, FeTiO<sub>3</sub>.nFe<sub>2</sub>O<sub>3</sub>; others again correspond to neither. Snelling explains this variable composition as follows:<sup>59</sup>

Both ferric oxide and titanium oxide unite with ferrous titanate (ilmenite) in all proportions, forming a series of compounds varying continuously in composition between the limits  $TiO_2$  and  $Fe_2O_3$ . Thus we have light yellow rutile consisting of practically pure titanium oxide; darker coloured rutile containing a small quantity of  $FeTiO_3$ ; then black rutile (nigrine) containing a relatively greater proportion of  $FeTiO_3$ , and so on, until the ferrous titanate becomes the principal constituent and the proportion of  $TiO_2$  becomes less and less. But the series is extended still further, for the  $FeTiO_3$ , combining with  $Fe_2O_3$ , produces compounds in which the ratio between the  $Ti_2$  and  $Fe_2$  becomes fractional, and which contain a greater quantity of  $Fe_2O_3$  than  $FeTiO_3$ ; and this continues until we have  $Fe_2O_3$ , containing a very small quantity of ferrous titanate, the compound now representing a slightly titaniferous hematite.

The compounds intermediate between rutile and hematite, are, collectively, called titanic iron and include, besides ilmenite proper, such sub-species as nigrine, ilmenorutile, kibdelophane, crichtonite, hystatite, uddevalite, and basanomelan, etc., in which the percentage of titanium lies between certain arbitrary limits and which do not represent true minerals, but mineral mixtures.

Studying the composition of the ilmenite from St. Urbain, Dr. Warren<sup>60</sup> arrives at the conclusion that it is an intimate crystallographic intergrowth of ilmenite and hematite, and that the ilmenite and hematite molecules are not isomorphous as is so commonly reported for other localities. In a later paper "On the microstructure of certain titanic iron ores" he finds that complete solid solution occurs between ilmenite and hematite at higher temperatures, with an inversion interval and slight miscibility at lower temperatures, and suggests that molecules of Ti<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> may enter into solid solution as such.

The following are analyses of some typical ilmenites from different parts of the world:--

#### Analyses of Ilmenite

	1	2	3	4
ΓίΟ <sub>2</sub> FeO	Per cent $54 \cdot 44$ $40 \cdot 94$ $4 \cdot 26$ $0 \cdot 34$ $0 \cdot 52$ $0 \cdot 07$	Per cent 41.75 31.01 22.11 0.60 0.28	Per cent 40·00 29·57 20·35 1·91 4·00	50·92 39·40 9·17
Al <sub>2</sub> O <sub>3</sub>	0.29	$\begin{array}{c} 3 \cdot 15 \\ 0 \cdot 55 \end{array}$	$3.17 \\ 1.00$	
$egin{array}{cccccccccccccccccccccccccccccccccccc$	Trace 100.86	99.95	100.00	99.61

- 1. Ilmenite from Nelson county, Virginia; General Electric Co.'s mine. (Watson and Taber).
- 2. Ilmenite from Ekersund-Soggendal, Norway (Vogt). 3. Ilmenite from St. Urbain, Quebec (Adams).
- 4. Ilmenite from Nelson county, Virginia; American Rutile Co.'s quarries (Watson and Taber).

According to Lindgren,<sup>54</sup> and Emmons,<sup>55</sup> ilmenite is formed in igneous rocks of all kinds, in pegmatites, in contact metamorphic deposits, under regional metamorphic conditions, and in deep veins. As a rock mineral it closely resembles magnetite and hematite, with which it is commonly associated. The most important occurrences of ilmenite are in the coarser grained gabbros and anorthosites, where it is not infrequently found segregated locally into large masses.

Vogt<sup>45</sup> finds that iron oxide segregations occurring in gabbro, labradorfels (anorthosite), and, less frequently, in augite and nepheline syenites, are characterized, without exception, the world over, by a considerable titanium content (ilmenite), the titanium rarely falling below four or five per cent in the segregations rich in iron. Those occurring in anorthosite relatively poor in ferromagnesian minerals are usually the more concentrated and are characterized by a higher titanium content than those found in the darker gabbros. In addition to titanium, vanadium is usually present in small amounts and, occasionally, a little chromium, nickel, and cobalt. Phosphorus and sulphur are usually, but by no means invariably, low.

These titanium-iron segregations are not peripheral deposits, but occur in the interior portions of eruptive masses. Some of them, including most of those occurring in the darker gabbros, pass gradually, by diminution of the titanium-iron oxides and increase of silicate minerals, on all sides of the central core to normal rock, a mode of occurrence to be explained by their differentiation in situ. Others, chiefly those associated with anorthosite, occur as irregular masses or dike-like bodies, showing sharp contacts with the adjacent rock. Differentiation in this case must have taken place at depth, after which the resultant iron-titanium magma was extruded, like any ordinary diaschist rock, through the already solidified mass.

Canadian occurrences of titanium-iron oxides conform, both in composition and mode of occurrence, to the general characteristics of this class of deposit

as described by Vogt. Those at Ivry and St. Urbain, in Quebec, are the only ones to be classed as ilmenites (in contradistinction to titaniferous magnetites) and at present known to be of prospective commercial importance. The ilmenite at both these places is found in comparatively pure masses, showing sharp contacts with the anorthosite in which they occur. Excepting certain portions of one of the St. Urbain deposits that are enriched by rutile, they carry from 30 to 40 per cent of titanium dioxide, at least traces of vanadium, and are comparatively low in sulphur and phosphorus.

Practically speaking, ilmenite may be described as titanic iron containing 40 per cent and upward of TiO<sub>2</sub>. Such material is sold as titanium ore, at a price based on its titanium content. It is used almost entirely for the manufacture of ferrotitanium alloys in the electric furnace. By fractional reduction in the electric furnace, titanic acid, TiO<sub>2</sub>, may be obtained from it.

#### TITANIFEROUS MAGNETITE

Just as some ilmenites contain intimately intermixed hematite, so titaniferous magnetites consist in large part of intimate intermixtures of magnetite and ilmenite, and in so far as they consist of such mixtures may be regarded as special occurrences of ilmenite. There is evidence, however, that at least some of the titanium in titaniferous magnetites does not occur as ilmenite but chemically combined in the magnetite molecule itself, where it replaces iron (titanomagnetite).

Singewald<sup>65</sup> who studied the question of the composition of titaniferous magnetites very thoroughly, chiefly by metallographic methods, concludes that the titanium occurs partly in the form of ilmenite grains mixed with magnetite grains of about the same order of magnitude, partly as ilmenite inclusions and intergrowths of microscopic size in the magnetite, and partly as an integral part of the magnetite molecule itself, the proportions occurring in these different forms varying greatly in different ores. Crystallographic intergrowths of rutile in magnetite are also known to occur. Warren<sup>61</sup> says there appears to be some solid solution of ilmenite and magnetite molecules, and also a cutectic; in some cases rutile forms where there is an excess of TiO<sub>2</sub>.

Titaniferous magnetites show a wide range in their titanium content. Small traces of titanic acid can be detected in almost all the magnetites in the old crystalline gneisses, but ores are not usually described as titaniferous unless 3 per cent or more of titanic acid is present. From this minimum all percentages are recorded up to 50 or more, 63 but few go much above 25 per cent; and for practical purposes that may be regarded as the upper limit for titaniferous magnetite; all carrying more than 25 per cent TiO<sub>2</sub> being regarded as ilmenites, and essentially titanium ores.

In other words, ilmenite, or titanic iron, includes, in addition to ilmenite properly so called, those high titanium, non-(or only feebly)magnetic minerals consisting of the oxides of titanium and iron whether in chemical combination or intimate mechanical intermixture; while titaniferous magnetite includes those distinctly magnetic minerals, consisting of the oxides of the same two elements, in which a part of the iron is in the form of the magnetic oxide, Fe<sub>3</sub>O<sub>4</sub>. It is in this way that ilmenite, or titanic iron, is distinguished from titaniferous magnetite throughout this report.

In their general mode of occurrence and mineralogical associations there is little to discriminate titaniferous magnetites from ilmenites, into which they grade. In Canada, titaniferous magnetite is of more frequent and more wide-spread occurrence than is ilmenite, and is usually associated with gabbro rather than anorthosite. Bodies, practically of solid mineral, may attain considerable size, but usually they are small and scattered through ferromagnesian rich phases of the gabbro into which they pass gradually on their borders. Where, as not infrequently happens, the immediately surrounding rock assumes the form of black pyroxenite or peridotite, perhaps with a little disseminated magnetite scattered through it, the distinction between rock and ore is not always readily perceptible, 63 and casual inspection may lead to exaggerated estimates of the extent of the actual ore-body, especially when outcrops of different segregations lie close together. If we are to judge by still current descriptions of them, something of this sort appears to have happened in the earlier examinations of some of our Canadian occurrences of titaniferous magnetite.

It is sometimes possible to distinguish non-titaniferous magnetite from the titaniferous variety in hand specimens, especially if the mineral is coarse-grained, by the peculiar triangular striae often developed on the cleavage faces of the latter. These striae are produced by lamellar intergrowths of ilmenite, in the form of thin tablets intercalated in the magnetite parallel to its octahedral faces. In other, platy, specimens, when broken across the lamellae, the edges of intercalated ilmenite leaves appear as fine, bright, parallel lines against a dull black background, giving the mineral a streaked appearance.

#### Blast Furnace Smelting of Titaniferous Magnetites

Titaniferous magnetite as defined above, that is, magnetite carrying from 3 to 25 per cent titanium is at present a material economically undesirable for any purpose. It is too low in titanium to find favour as a source of that metal in competition with the richer ilmenites, whereas the titanium content renders it anathema to the modern blast-furnace man even when it has otherwise all the qualities requisite in a good iron ore. The objection made to its use by iron smelters is not that the iron made from it is inferior in quality—irons made from titaniferous ores are admittedly better, if anything, than other irons—but that it makes a pasty slag; that it forms accretions, causing scaffolding in the furnace and clogging in the hearth; and that the fuel required for its reduction is excessive in comparison with that necessary for the reduction of non-titaniferous ore. Nevertheless it has been used, both knowingly and unknowingly, and apparently without difficulty, for the making of iron in the past, and in the opinion of many, it is as an iron ore that it will find a use in the future.

To illustrate the former use of titaniferous ores in the manufacture of iron, a number of instances may be cited. A century ago fifteen, and in 1875, ten small blast furnaces were in operation at Taberg, 45 Sweden, on ores carrying 6 or 7 per cent titanic acid, and the production of iron from titaniferous ores had not entirely ceased in Sweden in 1904. The consumption of fuel in the larger Swedish furnaces, however, is said to have averaged 275 bushels of charcoal per ton of ore smelted, a quantity considerably in excess of that required for non-titaniferous ores. 45, 64, 65, 58

For several years, about 1868, The Norwegian Titanic Iron Company smelted a highly titaniferous ilmenite at Norton, near Stockton-on-Tees, in England. Coke was used for fuel and old bricks and similar material, in addition to limestone, for flux. The ore contained only 35 or 36 per cent iron and 38 to 40 per cent titanic acid; the slag carried 36 per cent titanic acid. The process is described as technically a success, but, owing to the uncertainty of the ore supply, its low tenor in iron and the consequent abnormal consumption of fuel (nearly three tons for each ton of iron made), operation was not profitable. 16,66

David Forbes<sup>67</sup> smelted ores containing 7 per cent and 15 per cent titanic acid, using charcoal for fuel, and, after experimenting first with quartz and then with limestone, finally adopted a mixture of limestone and quartz for flux. The consumption of charcoal was about 20 per cent in excess of normal requirements, but the ores contained only 39 and 42 per cent respectively of iron. Forbes found the furnace worked satisfactorily provided there was not more than 8 per cent titanium in the ore.

At Sanford lake, in New York state, two small charcoal furnaces, one of 4 tons and the other of 15 tons daily capacity, were successfully operated from 1840 to 1856, on ores carrying 8 to 15 per cent titanic acid and were only abandoned on account of their inaccessible location, which made the cost of transporting the pig iron to market excessive. 16, 17

In 1873, two charcoal blast furnaces were erected in the parish of St. Urbain, north of Bay St. Paul, Quebec, to manufacture iron from the ilmenite found there, which carried about 25 per cent titanium and 45 per cent iron. They were in blast but a few months, during which they appear to have operated under somewhat adverse conditions. Good pig iron was made, but the consumption of charcoal never fell below 3,040 to 3,790 pounds per ton of pig iron produced.<sup>68</sup> The quality of the pig iron made is shown by the following analysis, quoted by Riley in the Journal of the Iron and Steel Institute for 1874 (p. 132). It may be noted that no titanium appears in the iron in spite of the high proportion of that metal in the ore.

#### Analysis of Pig Iron made at St. Urbain, Quebec

Carbon	3.966	3.976
Silicon	0.086	0.081
Sulphur	0.030	0.048
Phosphorus	0.253	0.258
Iron.	95.245	95.440
Chromium	0.689	0.436
Manganese	Trace	Trace
Titanium		
	100.269	100.239

In 1867, the Moisie Iron Company built eight bloomery furnaces at Moisie, on the north shore of the St. Lawrence, for the production of iron by the direct method from the titaniferous iron sands found there. The sands were first concentrated on shaking tables and by a process of magnetic separation to a product containing probably 65 or 70 per cent of iron and less than 2 per cent titanium dioxide; but before going to the furnace the titanium content was sometimes considerably increased by adding to the concentrates highly titaniferous magnetite brought from Bay of Seven Islands. In just

what proportions the two materials were mixed is not recorded. The furnaces made about one ton of iron and consumed 6,990 pounds of charcoal per day each. The iron contained no titanium, but is said to have been of excellent quality, equal to the best Swedish and suitable for the manufacture of the finest steels.

A portion of the blooms produced was shipped to Montreal to be worked into forgings, etc., but the chief market for them was in the United States. When, in 1875, the United States Government ruled that the blooms were to be classed as bar iron, subject to a duty of 1½ cents per pound, instead of as pig iron liable to a duty of 7 per ton, as heretofore, this market was closed. As a consequence, the works were shut down and the company went into liquidation. 69, 70, 71 It may be noted that the iron made at Moisie was not pig iron but wrought iron, by the direct process, a method of manufacture that is now obsolete.

Bowron, 66 as the result of his experience in smelting titaniferous ores, concludes that:—

The whole secret of working these ores successfully and continuously is to keep the heat so low as just to reduce the iron and not reduce the titanic acid. The iron will be white, or at best mottled, if there is much titanic acid to contend with. Titanic iron is essentially a forge iron. Foundry iron can only be produced when the titanium is low, and then only by making a large quantity of cinder, so as to wash the titanic acid out of the furnace.

David Forbes,<sup>67</sup> who was for some years connected with charcoal iron works in southern Norway, where titaniferous ores were used, says that in the experience of the Scandinavian ironmasters the only objection to their use is the excessive amount of charcoal required for their reduction, and, hence, their use is not profitable where non-titaniferous ores are available. For this reason he found it unprofitable to smelt alone an ore carrying 42.04 per cent iron and 15.10 per cent titanic acid, but could readily use such an ore mixed with an equal quantity of titanium-free ore. He also found that by using mixed quartz and limestone as a flux, satisfactorily fluid slags could be made, and that when the titanium in the ore did not exceed 8 per cent no difficulty was experienced in working the furnace cleanly and profitably.

Passing on to the more recent investigations of Rossi and Bachman. In 1890, Rossi<sup>15</sup> published a review of what had been accomplished in the past in the smelting of titaniferous iron ores. He followed this up, three years later, with an account of some experiments he had conducted along the lines adopted by Forbes, in an attempt to discover easily fusible titanate slags of low viscocity. His experiments were, at first, conducted in a small furnace without forced draft, later in a small blast furnace. In the latter, with ores ranging from 18.70 to 20.03 per cent titanic acid, he obtained pig iron and good, fluid, easily fusible slags. The fuel used was coke and the flux dolomitic limestone, with the addition of broken bricks and quartz at the beginning of operations. In 1895 he repeated his experiments on a larger scale, in a furnace having a daily capacity of three tons, with similar results.

Summing up the results of his experiments, he concludes:<sup>17</sup> that titanic acid if properly fluxed forms perfectly fusible compounds containing as much as 30 to 50 per cent TiO<sub>2</sub> with alumina, lime, and magnesia as bases. These compounds are in every way suitable as slags in blast furnace work. The objection to the smelting of titaniferous ore on account of the refractory character of the slags is not sustained by the experiments.

Running the furnace under special conditions of pressure and temperature of blast, no troubles were experienced from titanium deposits.

In a later paper he says: no difficulty will be experienced in smelting if the oxide of titanium (TiO<sub>2</sub>) is regarded as so much silica and sufficient limestone is added to flux it off, and the slag will be more fusible if dolomite is used instead of limestone. As regards the pig iron made<sup>14</sup>:—

It is essentially an open-hearth stock. Analyses of the pig iron made by us and that of the Adirondacks (the same ores were used in both cases) showed: phosphorus, traces; silicon, 0.11 to 0.13; carbon, from 1.86 to 3.50 per cent, practically all combined; the fracture had very much the appearance of that of steel. It is eminently a chilling iron, well adapted for car wheel mixtures.

Perhaps the most convincing experiment yet made in the smelting of titaniferous iron ores was that carried out on a large scale in a modern furnace in 1914, under the direction of F. E. Bachman, general manager of the McIntyre Iron Company (the McIntyre Iron Company are the owners of a large deposit of titaniferous magnetite in the Adirondacks). The test was made at Port Henry, New York state, in a furnace belonging to the Northern Iron company capable of producing 200 tons of pig iron per day, and was extended over a period of more than two months, during which time several thousand tons of titaniferous ore was smelted. The results, therefore, can be accepted as a reliable indication of what may be expected in commercial operation. A full account of the investigation has been published by Mr. Bachman in the Year Book of the American Iron and Steel Institute for 1914.<sup>72</sup>

In normal operation, the furnace in which the test was made is run on a mixture of all magnetite non-titaniferous concentrates with coke as fuel. For the purposes of the experiment from one-eighth to five-sixteenths of the normal ore charge was replaced by an equal weight of titaniferous-magnetite concentrates, the composition of which is indicated by the following analysis:—

#### Analysis of Sanford Concentrates

Iron	55.95
Silica	2.55
Phosphorus	0.003
Manganese	0.18
Alumina	3.98
Lime	0.12
Magnesia	1.62
Titanic acid	11.80
Vanadic oxide	0.40
Chromium oxide	0.244
Nickel oxide	0.049
Sulphur	0.119

Using one-eighth titaniferous concentrates, a foundry iron was made without any serious difficulty and with a normal consumption of fuel. The fluxes consisted of dolomite and marble (calcite). The iron contained 0.5 per cent titanium and about 0.06 per cent vanadium; the slag carried about 4 per cent titanic acid.

With about one-fourth titaniferous concentrates in the ore charged, malleable iron was made, that is, pig iron low in silicon, suitable for the production of malleable castings. This iron contained about 1 per cent silicon and 0.45 per cent titanium, while the slag carried about 6 per cent titanic acid.

Neither the output of the furnace nor the consumption of coke per ton of iron produced was materially affected by the substitution of titaniferous for

titanium-free concentrates. The results of the experiment go to show that titanium does not form infusible slags nor deposits in the furnace when suitably fluxed; that as little as 2 per cent titanium in the ore may interfere with the production of foundry, or high silicon, iron; that titanium behaves like silicon in the furnace but is less acid; and that under the conditions of high temperature and abundant fuel necessary to produce a high silicon or foundry iron, titanium is reduced and enters the iron somewhat to the exclusion of silicon. The iron made from titaniferous ores would appear to be specially suited for the production of steel in the open hearth furnace and for use in chilled iron and similar castings.

A recent experiment in the smelting of titaniferous magnetic iron sands, in New Zealand, has been briefly described by J. A. Heskett.<sup>73</sup> The experiment was carried out in a small blast furnace, with hot blast, using coke as fuel. The output averaged 5 tons per day, and, in all, about 50 tons of iron were made. The slags were fluid, even when they contained as much as 20 per cent titanic acid, and no difficulty was experienced in their handling; indeed, the results would lead to the belief that the titanium content had lowered their fusion point as well as their viscosity. The iron produced was exceptionally strong and tough. With 2.5 per cent silicon and 0.04 per cent sulphur, it showed a tendency to chill even in ordinary sand casting.

Accounts of other experiments in the smelting of New Zealand titaniferous iron sands are given in publications of the New Zealand Department of Mines. (See Bull. 14, new series, Geol. Sur. Branch, Dept. of Mines, New Zealand, 1912,

pp. 48-51; and N. Z. Mines Statement, 1914, p. 26.)

It would appear from a review of the records that earlier in the history of the iron industry—in the days of the Catalan forge and the small charcoal blast furnace—when ironworks were located in well wooded districts, close to their supplies of fuel and ore, it made little difference whether the ores used were titaniferous or not. Having a cheap and plentiful supply of charcoal, the fuel consumption was a matter of no great moment so long as good iron was made; and good iron could be and was made from titaniferous ores, apparently without any special difficulty and in some instances without the titaniferous nature of the ore being even suspected.<sup>74</sup> Whether the good quality of the iron thus produced was due to the presence of titanium in the ore or had its origin in the usual comparative freedom of such ores from sulphur and phosphorus need not be discussed here, suffice to say that this is a point on which the opinion of metallurgists differ. Later, with the increasing size of blast furnaces, and charcoal becoming scarcer and more costly as the forests were depleted, economy of fuel became a matter of much greater importance than it had heretofore been. At the same time little attempt appears to have been made to adapt the furnace charge to the particular nature of the ore used, and as the slags produced from titaniferous ores in the ordinary blast furnace practice were viscid and had to be run at a higher temperature than ordinary slags, conditions necessitating not only a high fuel consumption but probably favourable also to the formation of infusible titanium compounds in the furnace, there would consequently arise a prejudice against their use. Moreover, the use of titaniferous ore would mean the use of a lower grade ore; since the titanium replacing its equivalent of iron in the ore means so much more worthless material to be fluxed and more slag to be melted, and this also would increase the fuel consumption. As a consequence, during the evolution of modern blast furnace practice titaniferous ores fell into disrepute, their use gradually declined, and finally ceased altogether.

The experiences of Rossi, Bachman, and other investigators, all seem to demonstrate that the commonly cited objections to the use of titaniferous ores, viz., viscid slags, clogging of the furnace, and excessive fuel consumption, can be overcome (at least in the case of ores containing only a moderate amount of titanium) by changing the furnace practice to suit the ore, that is to say, by the maintenance of suitable temperatures and pressure of blast, and by the use of proper fluxes.

The influence of titanium in forming accretions in the furnace, however, appears to be cumulative in its effect<sup>85</sup> and might only become clearly evident after longer runs than those made by Rossi and Bachman. In ordinary furnace practice they appear to form in time even when the titanium content is very small.

An experienced operator and well known authority on the blast furnace, J. E. Johnson, Jr., commenting on the use of titaniferous ores, in his book on the "Principles, Operation, and Products of the Blast Furnace", says: (p. 28),

There is much reason to believe that the former verdict against it (titanium) was on insufficient grounds," and (p. 258) "there seems to be no longer any reasonable doubt that it is quite possible to operate furnaces successfully on ores containing amounts of titanium which at one time were firmly believed to be prohibitive.

Nevertheless, no attempt is being made to smelt such ores, and there is no very hopeful feeling among iron smelters in regard to their commercial possibilities. Deposits of titaniferous iron ore are, as a rule, small in extent and irregular in their mode of occurrence; furthermore, they are low, or only of medium grade, in iron; and few of them are advantageously situated with respect to existing iron making centres. To make assured from them the large and continuous supply of ore necessary to keep in operation a modern iron making plant would involve an expenditure not warranted by the outlook. Operators, moreover, have an entirely justifiable disinclination to turn from established practice, based on adequate known supplies of satisfactory material, to one that does not offer any obvious additional advantage, and the economic limitations of which have yet to be determined. The opinion of another iron master on this phase of the question is worth considering. Discussing a paper read before the American Institute of Mining Engineers, on some magnetic iron ores occurring in North Carolina, B. F. Fakenthal, Jr., of the Durham Iron Works, Riegelsville, Pa., says<sup>75</sup>:—

The high titanium ores, so far as I know, usually carry less than fifty per cent of iron and the gangue that must be fluxed in smelting them is certainly more difficult to deal with than an equal proportion of ordinary earthy gangue, or even of silica. The cost of mining such ores is not less than that of mining other hard magnetites. It seems to me that there is no very strong demand for a special invention to overcome the difficulty of smelting a material which would not be desirable even if there were no such technical difficulty, because it does not yield iron enough to pay for mining, transportation, flux, fuel, and furnace labour under average conditions. It is possible to make pig iron from titaniferous ores, but it is not profitable, and the reason it is not profitable, apart from the refractory character of the ores, is their poverty.

#### Electric Smelting of Titaniferous Iron Ores

Where conditions are favourable, as in Sweden, the electrothermic smelting of iron ore has had considerable commercial success, and it has been thought by some that this method of reduction would prove particularly adapted to

titaniferous ores. This opinion was based largely on the belief that infusible slags would be produced in any attempt to smelt such ores in the blast furnace, and that the higher temperatures available in the electric furnace would overcome the difficulty; whereas, it has been shown that titaniferous slags are easily fusible and that the ores can be smelted at moderate temperatures. It may be further said that for commercial success in electric smelting it has so far been found necessary to use only the higher grades of iron ore, whereas titaniferous ores are usually low in iron and would have to be subjected to an expensive process of crushing and concentration to bring them up to the grade of ore used in the Swedish furnaces.

Pig iron has been produced experimentally in the electric furnace from titaniferous ores, as described by Haanel, <sup>76</sup> Gin, <sup>41</sup> Borchers, <sup>39</sup> and others, and its production in this way presents no special technical difficulties. (Borchers' investigations, be it noted, carried on for over twelve years, had in view the dual object of reducing the iron and recovering the titanium.) The question of the relative merits of electric versus blast furnace smelting of titaniferous ores does not appear to differ in any essential respect from the same question in regard to titanium-free ores; the arguments for or against either method in one case would be equally potent in the other.

Some experiments on the smelting of titaniferous iron ores in the electric furnace by W. M. Goodwin<sup>88</sup> indicate the possibility of obtaining satisfactory

slags by using silica instead of lime as a flux for such ores.

The production of high grade special steels direct from titaniferous ore in the electric furnace affords perhaps better chances of commercial success than the production of electric pig iron. Experiments carried out by Evans 77, 78 and Stansfield 79, 80, 81 showed that an excellent quality of steel, suitable for tool steel and steel castings, can be obtained in this manner at a moderate cost. There do not appear to be any technical limits to the quantity of titanium allowable in ores for this process, but the cost of smelting will of course increase with the quantity of titanium, both on account of the cost of fluxing it and on account of the smaller proportion of steel obtained. This will probably limit the commercial application of the process to ores containing less than 10 per cent of titanium.

Any advantage there may be in smelting titaniferous iron ores in the electric furnace will be shared equally by non-titaniferous ores unless it can be shown that the presence of titanium is of itself advantageous in the process; otherwise electric smelting will not make any change in the relative desirability of these two classes of material for making iron.

### Magnetic Concentration of Titaniferous Magnetites 62, 63

That many titaniferous magnetites consist, in part at least, of a mechanical mixture of ilmenite and magnetite can be seen in many cases on careful examination even with the unaided eye; the highly lustrous rougher surfaces of the ilmenite being easily distinguishable from the duller black cleavage faces of the magnetite (the contrast between the two minerals is accentuated if the specimen be ground down to a smooth polished surface and immersed for a time in concentrated HCl; under these conditions the bright ilmenite retains its lustre, whereas the magnetite becomes a still duller black) and they have in the past been quite commonly regarded as mechanical mixtures simply, and

the belief was general that magnetic concentration would remove the non-titaniferous magnetite and leave behind the inert ilmenite. If all the titanium present were in the form of granular intermixed ilmenite, it should be possible to obtain a titanium-free concentrate by subjecting the crushed material to a process of magnetic separation, and attempts have been made to solve the problem of the utilization of titaniferous magnetites for the production of iron by eliminating their titanium content in this way; but such experiments have led only to disappointment. Though it is often possible to obtain a prothat lower in titanium than the untreated mineral, it has been found practically impossible to obtain one that is altogether titanium-free. The inference to be drawn from the results of experiments in magnetic separation is that the association of iron and titanium in titaniferous magnetite is not as simple as might be inferred from the megascopic appearance alone of the mineral; and is confirmed if specimens are examined under the microscope by the metallographic method. Examined in this way it will be seen that only a portion of the ilmenite occurs in the form of intermixed granules; another portion musists of minute inclusions and intergrowths in the magnetite itself with which they are so intimately associated in such a fine state of division as to render me peless any attempt at mechanical separation. Moreover, chemical investigations of apparently perfectly homogeneous crystals of titaniferous magnetite give ground for the belief that still another portion of the titanium occurs chemically combined in the magnetite molecule, as titanomagnetite. such circumstances a complete separation by mechanical means into titaniumbearing and titanium-free components is a practical impossibility. The proportion of the titanium occurring in the separable (granular) and in the inseparable (microscopic inclusions and intergrowths, and chemically combined in the magnetite) forms respectively varies greatly in different occurrences and must be determined experimentally for each individual case; about the best that can be expected from any titaniferous magnetite is that it will yield a product low enough in titanium to be used in the blast furnace in admixture with non-titaniferous material.

The results of a number of experiments in the magnetic separation of different Canadian titaniferous magnetites are given further on in this report, in connection with the descriptions of individual occurrences.

#### Vanadium in Titaniferous Magnetites

One of the most characteristic features of titaniferous iron ores is the presence in them of vanadium in small amounts, usually less than one half of ne per cent, or about 5 to 10 pounds of vanadic acid to the ton. In just what form it is combined in the iron ores is not known. In this connection it is interesting to note that the element vanadium was first definitely recognized as such in pig iron made from the titaniferous ores of Taberg, Sweden. 45, 62, 63, 82

Vanadium is also found in iron ores that are not known to be titaniferous, such as the Minette ores of France and other French ores. For a time in the last century the sole commercial source of vanadium was the slags produced at the Creusot Steel Works, in France.<sup>83</sup> The slags from the basic hessemer converters at Creusot, which used the pig iron obtained by the reduction of oolitic ore from Mazenay, contained over one per cent of vanadium,

<sup>1</sup> The basic bessemer process was introduced into France about 1880 46410—3

and at one time the production of vanadium salts by the Creusot works represented about 60,000 kilos of vanadium annually.<sup>82</sup> In small amount, vanadium is of not unknown occurrence in pig irons derived from other ores not known to be titaniferous. It is found in many deposits of iron which have been formed by hydrochemical processes.<sup>45</sup>

When vanadiferous iron ores are smelted in the blast furnace, in ordinary practice part of the vanadium goes into the slag, but considerable fractions of one per cent may pass into the iron; little or nothing is known concerning its control. Bachman, 12 in his experiments on smelting titaniferous iron ores at Port Henry, New York, found that the vanadium content of the iron produced was more nearly proportional to the amount charged in the ore than in the case of titanium, but was not entirely so. Goodwin found that by smelting titaniferous iron ore in the electric furnace and using silica instead of lime as the flux it is possible to reduce 90 per cent of the vanadium with the iron.

In the process of converting pig iron into steel any vanadium originally present in the pig iron is completely eliminated in the slag.

The presence of vanadium in titaniferous iron ores has become of special interest since the element has come into such extended use in the manufacture of high grade alloy steels; for though vanadium has a remarkably wide distribution in the earth's crust, deposits rich enough to be of commercial value are rare, and some have looked to the titaniferous iron ores as a possible source of an augmented future supply. Over forty years ago an unsuccessful attempt was made to produce vanadium commercially from the titaniferous magnetite of the Church mine, in western New Jersey, and quite recently the technical press (The Mining Journal, Jan. 1, 1921, p. 14) records the formation of a company in Sweden to recover vanadium from the old blast furnace slags at Taberg. In Canada, investigations were recently undertaken, under the auspices of the Advisory Council for Scientific Research, with a view to the economic recovery of vanadium from titaniferous ores in the electric furnace.

Having regard to the interest at present taken in vanadium, there have been made in the Mines Branch laboratory, by Mr. H. A. Leverin, a number of determinations of the vanadium content of different Canadian titaniferous ores. These, supplemented by other analyses from various sources, are set forth in the following table:—

# Partial Analyses of Specimens of Canadian Transforms from Ores

Remarks	Ilmenite.  Magnetite  """  """  """  """  """  """  """	23
Analyst	H. A. Leverin (a)  """""""""""""""""""""""""""""""""""	H. A. Leverin (a)
Z	Trace Nii Nii Nii Nii Nii Nii Nii Nii Nii Ni	Z
CrgO <sub>s</sub>	0.25 0.32 0.08 0.08 0.08 0.07 Nil Trace Nil Trace 0.11 0.05 1.12 0.05 1.13 0.05 0.05	Trace
1.00	Trace 0.04 0.07 0.10 0.10 0.10 0.12 0.13 0.13 0.10 0.10 0.10 0.10 0.10 0.10 0.10 0.10 0.10 0.10 0.23 0.10 0.10 0.23 0.10 0.10 0.10 0.23 0.10 0.10 0.23 0.10 0.10 0.10 0.25 0.10 0.25 0.10	0.29
Δį	0.148 0.144 1.315 0.034 1.315 0.010 0.010 0.010 0.082 0.082 0.010 0.082 0.082 0.011 0.082 0.082 0.012 0.0137 Trace 0.082 0.012 0.0137 Trace 0.082 0.012 0.014 0.016	0.062
2	0.058 0.076 0.076 0.076 0.039 0.039 0.039 0.039 0.039	0.385
C. Saning	25.55 25	58.82
3.00	20.046 33.0706 33.0706 33.0706 33.0706 33.0706 34.0707 35.0707 36.0707 37.0	6.79
Locality	1. Furnace mine, St. Urbain, Charlevoix co. 2. Can. Gen. Elec. Co. Smine, St. Urbain, Charlevoix co. 3. Ivry mine, Terrebonne co. 4. Degrosbois mine, Terrebonne co. 5. St. Charles mine, Chicoutimi co. 6. Drill core, mining claims A.L. 26-£7, Rainy River district. 7. Mining claim A.L. 26, Rainy River district. 8. Mining claim A.L. 26, Rainy River district. 9. Mining claim H.P. 96, Rainy River district. 10. Lot 136, Lount tp., Parry Sound district. 11. Lots 143-144 Lount tp., Parry Sound district. 12. Matthews mine, North Crosby tp., Leeds co. 13. Chaffey mine, South Crosby tp., Leeds co. 14. Chaffey mine, South Crosby tp., Leeds co. 15. Chaffey mine, South Crosby tp., Leeds co. 16. Eagle Lake, Hinchinbrooke tp., Frontenac co. 17. Eagle Lake, Hinchinbrooke tp., Frontenac co. 18. Pine Lake or Pusey mine, Haliburton co. 19. Pine Lake or Pusey mine, Haliburton co. 20. Lot 30, con. xiii, Dungamon tp., Hastings co. 21. Lot 16, con. "A.", Faraday tp., Hastings co. 22. Lot 54, Hastings rd., Tudor tp., Hastings co. 23. Orton mine, Tudor tp., Hastings co. 24. Orton mine, Tudor tp., Hastings co. 25. Lot 54, Hastings rd., Tudor tp., Hastings co. 26. —, Horton tp., Renfrew co.	27. Near Burmis

(a) Mines Branch laboratory, Ottawa
(b) Walz, Isidor, On the general occurrence of vanadium in American magnetites: American Chemist, June, 1876, p. 453. Quoted by J. F. Kemp in the 19th Ann. Report U.S. Geol. Sur., Pt. 3, 1897-98, p. 392.
(c) Fope, F. J., Investigation of magnetic iron ores from eastern Ontario; Trans. Amer. Inst. Min. Figrs., vol. 29, 1899, p. 380.
(d) Evans, J. W., Tool steel from titaniferous magnetite; Jour. Can. Min. Inst., vol. 15, 1912, p. 128.
N.B.—The Mines Branch analyses were made on selected samples chosen for their freedom from rock matter and not as representing the deposit as a whole.

46410-31

It has been suggested that there is a general relationship between titanium and vanadium in titaniferous iron ores, and Pope, from a study of the analyses of five eastern Ontario magnetites and as many from the Adirondacks, arrives at an approximate ratio of vanadic oxide to titanic oxide in titaniferous magnetites of 1:28. Study of a more extended series of analyses than that on which Pope based his conclusions does not bear this out. For example, the Quebec ilmenites carrying 30 to 50 per cent TiO2 carry little better than traces of vanadium according to Mines Branch analyses; also, two samples of titaniferous magnetite from the Rainy River district, carrying respectively 19.87 per cent and 27.38 per cent TiO2, yielded in the first case only a trace of vanadium and in the second none at all, while a sample from the Orton mine carrying only 13.89 per cent TiO<sub>2</sub> yielded 0.34 per cent V<sub>2</sub>O<sub>5</sub>. Bachman,<sup>72</sup> also, reports that magnetic concentrates obtained from Sanford Lake (Adirondack) ore analysed: 56.29 per cent iron, 12.34 per cent TiO2, and 0.47 per cent V2O3; while ilmenite mechanically separated from the tailings from the same orc yielded: 34 13 per cent iron, 49 10 per cent TiO2, but only a trace of V2O3. A review of a wide range of published analyses shows no constant ratio of vanadium to titanium and indicates that vanadium is not characteristic of titaniferous iron ores alone. (See also analyses of magnetites from New Jersey and Minnesota given by J. T. Singewald, on pages 80 and 108 of U.S. Bureau of Mines Bull. 64.)

### **BIBLIOGRAPHY**

References to titanium are exceedingly numerous and widespread, and a comprehensive bibliography of the literature relating to it is entirely beyond the scope of this report. A numbered list of the authorities referred to in Part I of the report is given below, the numbers corresponding to those in the text. Further references will be found in the footnotes in Parts II and III.

Partial bibliographies of the literature of titanium are also to be found in numbers 1, 29, and 36 of the subjoined list.

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# PART II TITANIUM OCCURRENCES IN CANADA



### TITANIUM OCCURRENCES IN CANADA

All the known occurrences of titanium in Canada that are of any possible economic interest are in the provinces of Quebec and Ontario. Ilmenite, or titanic iron, in commercial quantity and carrying 20 to 30 per cent titanium, is found in two localities in Quebec, at St. Urbain in Charlevoix county and Ivry in Terrebonne county. There is enough rutile mixed with the ilmenite in parts of one of the St. Urbain occurrences to make it of possible importance for the rutile alone: this constitutes, so far as known, the only workable deposit of rutile in Canada. The rest of the Canadian titanium occurrences that are large enough to be of possible importance are all titaniferous magnetites, seldom carrying more than 15 per cent of titanium; those with less than 2 or 3 per cent titanic acid are not considered here, as such material has no possible value for its titanium content and is not debarred by its composition from use as an ordinary iron ore.

The five most important Canadian occurrences in point of known, or rather probable size, for they have not yet been delimited with completeness or great accurracy, are: ilmenite deposits at St. Urbain, and at Ivry, in Quebec; titaniferous magnetite deposits on the Saguenay river, near lake St. John, Quebec, those at Bay of Seven Islands, also in Quebec, and an extensive series found along the shores of Seine bay and Bad Vermilion lake in western Ontario. All are of the type characteristic of titanium-iron-oxide deposits the world over, that is to say, they are magnatic segregations genetically connected with basic eruptive rocks of the gabbro family and characteristically irregular and uncertain in their mode of occurrence.

Another Canadian deposit of a different type may also be mentioned here, not particularly on account of its titanium content, which is comparatively small, but on account of its possible future value as an iron ore. This is the beds of indurated magnetic black sands found in the foothills of the Rocky mountains near Burmis, Alberta. Analyses show that some of the material in these beds carries between 40 and 50 per cent iron with less than 4 per cent titanium, and that it is low in sulphur and phosphorus. Though material such as this would not generally speaking receive much consideration as an iron ore, its contiguity to coal fields and the lack of other iron ores in the district would give it a special interest should the quantity available prove to be very large.

## RUTILE OCCURRENCES PROVINCE OF QUEBEC

### Charlevoix County

Parish of St. Urbain.—The presence of rutile in considerable quantity in an ilmenite occurrence near St. Urbain, Charlevoix county, Quebec, was noted by T. Sterry Hunt in the Geology of Canada, 1863. No special attention appears to have been paid to it, however, until about 1910, when the General Electric Company of Schenectady, New York, who were at that time also interested in the development of rutile deposits in Virginia, U.S.A., acquired the

St. Urbain deposit by lease from the owners and, operating through a subsidiary the Loughborough Mining Company, opened up the deposit and shipped a little ore. The first detailed geological description of the occurrence was published by C. H. Warren, in the American Journal of Science, in 1912. Dulieux, in his report on the iron ores of the province of Quebec, issued by the Quebec Bureau of Mines in 1915, describes the ilmenite deposits at St. Urbain, but only incidentally mentions the occurrence of rutile in them.

The deposit is situated about nine miles by wagon road north of Baie St. Paul, a village on the north shore of the St. Lawrence river about 60 miles below Quebec, with which it is connected both by boat and rail, and is about two miles west of the little village of St. Urbain in the parish of the same name. The General Electric Company's workings, on the brow of the steep slope that, bounding the sandy valley of the Gouffre river on the west, overlooks the village of St. Urbain, are on lot 324 of the cadastral plan of the parish of St. Urbain and possibly 1,100 or 1,200 feet above tidewater at Baie St. Paul.

The country rock of the locality is an anorthosite, consisting chiefly of andesine and poor in ferromagnesian minerals. Its structure is for the most part massive, but in places it is gneissoid. Of the several ilmenite bodies that occur scattered through it in this vicinity the only one known to be rutile-bearing is that on the General Electric Company's property and that only in parts.

The workings, consisting of two shallow quarry faces having a maximum height of about fifteen feet, cut into the hillside, expose ilmenite over an area approximately 300 feet long by 90 feet wide. A borehole put down in 1911 is said to have proved the ore to a depth of at least 120 feet below the surface at the open-cuts. Where the contact between ore and rock can be seen in the workings, it stands nearly vertical and usually has a westerly trend. Feldspathic streaks included in the ilmenite body also appear to have a nearly vertical dip and a more or less distinctly banded structure, with the bands running east and west. This seemingly general westerly trend in the rock structure is repeated in several smaller dike-like masses of non-rutile-bearing ilmenite that have been exposed to the southwest of the main workings, but development work is not extensive enough to afford any conclusive evidence as to either the shape or attitude of the ore-body.

Excepting the rutile, the mineralogical composition of this mass of ilmenite is identical with that of the other ilmenite bodies found in the district. It consists of a dense black medium-grained ilmenite through which are scattered grains of andesine feldspar or its decomposition products, occasional grains of dark green spinel, and plates of dark brown mica, the accessory minerals constituting perhaps 2.5 to 6 per cent of the whole mass.

Certain portions of this mass, however, differ from the rest, and from other masses in the neighbourhood, in that they are rutile-bearing. These portions occur as indistinctly banded streaks and patches distinguished from the surrounding dead black ilmenite by the brownish tint given them by the intermixed rutile, and consist of rather finely granular ilmenite thickly sprinkled with orange-red rutile, a few feldspars, and a little biotite, spinel and sapphirine, the ilmenite forming the background in which the other minerals lie. The change from rutile-bearing to rutile-free ilmenite at their borders is usually quite sharp; within them the distribution of the rutile is not uniform. Microscopical examinations of thin sections show rutile present in some speci-

mens to the extent of 20 per cent, but it constitutes probably less than 6 per cent of the general run of the rutile-bearing material. Anorthosite (feldspathic) streaks included in the rutile-bearing portions of the ore-body also carry rutile, to the estimated extent of 2 or 3 per cent, accompanied by ilmenite and biotite arranged in parallel lines that produce a gneissoid effect. It was not found possible to arrive at any reliable estimate of what proportion of the whole mass is rutile-bearing.

The rutile-bearing ilmenite, like the other ilmenite bodies of the district, is regarded by Dr. Warren as a magmatic differentiation product in which there was an excess of titanic oxide and a smaller excess of magnesia and alumina, representing an extreme ultra-basic type of igneous rock derived from the same magma as the surrounding anorthosite.

Following are a number of analyses of the rutile-bearing ilmenite:-

### Analyses of Rutile-bearing Ilmenite from St. Urbain, Quebec

	1	2	3	4
iO <sub>2</sub> iO <sub>2</sub>	53·35 2·24	41·61 1·10	45·80 1·52	50.70
(nO	13.61 24.49 0.30			
gO aO	4·04 0·30	Two oo	0.04	0.10
20 5			0.04	0.00 Trace
r <u>o</u> Os				0.3
ontanium	$28.54 \\ 32.01$	44·52 24·98	37.73 $27.48$	$34.7 \\ 30.4$

<sup>1.</sup> Analysis of a rutile rich specimen, after Warren; Bull 580 U.S. Geol. Sur., 1915, p. 407.
2. Analysis of a general sample, after Dulieux; Les Minerais de fer de la Province de Quebec,
Quebec Bureau of Mines, 1915, p. 124.
3 and 4. Analyses made in the Mines Branch laboratory, Ottawa, by H. A. Leverin.

As has been said, the deposit was first opened up in 1910, by the General Electric Company, who still control it. Work was carried on during part of that and the following year and a few hundred tons of ore shipped to Schenectady, N.Y., to be used, it is said, in the manufacture of filaments for incandescent lamps and carbons for arc lights. Small shipments have been made at widely separated intervals since 1911, the last recorded being about 1,400 tons in 1918. The total amount of rutile ore sent out and the percentage of rutile in it is not known; it is returned simply as ilmenite, or titaniferous iron, and included in the published official statistics with other titaniferous ores under that heading.

There is no mining equipment of any kind on the property; all the work was done by hand and the ore hauled on carts or sleighs to Baie St. Paul for shipment.

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Hunt, T. Sterry, Geology of Canada, 1863, p. 501.

### MINOR RUTILE OCCURRENCES

### PROVINCE OF QUEBEC

### TEMPLETON TOWNSHIP

Previous to the exploitation of the St. Urbain deposits in 1910, the best known locality for rutile in Canada was in the township of Templeton, Hull county, Quebec. Some years ago considerable prospecting was done in this locality for rutile, which was reported to occur in some of the apatite veins of the district.

Harrington mentions its occurrence in small crystals in an apatite vein on lot 10, range X.

H. S. de Schmid says:—

Very large prisms (of rutile) occur in a barytes vein, associated with acicular, green actinolite, on range XIII, lot 13, N. 2, of Templeton. The vein occurs on the contact of amphibolite with crystalline limestone.

A boulder consisting of a similar association of minerals has been found in the adjoining township of Buckingham.

Johnston reports rutile in good crystals on range XI, lot 12, and on range XIII, lot 13.

References :-

Harrington, B. J., Geol. Sur. Can., Report of Progress for 1877-78, p. 356. de Schmid, H. S., Mica: its occurrence, exploitation, and uses, Mines Branch Pub. No. 118, 1912, p. 296. Johnston, R. A. A., A list of Canadian mineral occurrences, Geol. Sur. Can., Memoir 74,

1915, p. 195.

### Beauce County

In the auriferous sands and gravels of Beauce county, Quebec, the black residue left after washing, contains a large quantity of ilmenite, mixed with chromic iron, rutile, and magnetic and specular oxides of iron. Reference :-

Hunt, T. Sterry, Geology of Canada, 1863, p. 502.

### Brome County

### SUTTON TOWNSHIP

In the township of Sutton, Brome county, Quebec,

small red flattened crystals of titanic acid are found with chlorite, specular iron and orthoclase feldspar. It is difficult to determine whether they belong to the species rutile, or to brookite, both of which are pure titanic acid. Reference :-

Hunt, T. Sterry, Geology of Canada, 1863, p. 502.

### PROVINCE OF ONTARIO

### Algoma District

Rutile in the form of delicate acicular crystals lining drusy quartz cavities has been found in the Wallace mine, Bay of Islands, on the north shore of lake Huron.

Reference :-

Hunt, T. Sterry, Geology of Canada, 1863, p. 502.

### Hastings County

Rutile is found in tolerably distinct crystals on Green island in Moira lake, Madoc township, and in veins with chlorite in the adjoining township of Marmora.

References:-

Hoffman, G. C., Annotated list of the minerals occurring in Canada, Geol. Sur. Can., Ann. Report, vol. IV, 1888-89, p. 56T.

Geol. Sur. Can., Report of Progress for 1870-71, p. 311.

### PROVINCE OF NOVA SCOTIA

### Halifax County

A prismatic and massive form of black rutile—possibly nigrine, or ilmenorutile—has been identified in samples of auriferous quartz from the Irving vein, in the Mooseland gold district, Halifax county, Nova Scotia.

Hoffman, G. C., Geol. Sur. Can., Ann. Report, vol. 11, 1898, p. 16 R.

### Kings County

Transparent quartz penetrated with needles of rutile (sagenite, flesche d'amour, or Venus' hair stone) is reported from Scot's bay, King's county, N.S. Reference:—

Coste, Eugène, Geol. Sur. Can., Ann. Report, vol. 3, 1887, p. 70 S.

### YUKON TERRITORY

### Thistle Creek

Good specimens of a dark reddish-brown, sub-translucent, compact, massive rutile have been obtained from small quartz stringers traversing igneous schists on Thistle creek, a small stream entering the Yukon river some eight miles above the mouth of White river, Yukon district.

Reference:—

Hoffman, G. C., Geol. Sur. Can., Ann. Report, vol. 13, 1900, p. 21 R.

As a microscopic constituent of rocks of various kinds, rutile has been reported from a number of localities in Quebec, Ontario, British Columbia, and other parts of Canada. It is also found in small amount in the black magnetic sands that occur in various parts of the country.

### ILMENITE OCCURRENCES

As a minor rock constituent, ilmenite is of frequent occurrence in many parts of Canada, but workable deposits of this mineral, as distinguished from titaniferous magnetite, are found only in the province of Quebec, where large

deposits occur in two widely separated localities—St. Urbain in Charlevoix county and Ivry in Terrebonne county. It occurs at both places under similar geological conditions—in substantially pure deposits of various dimensions, distributed, apparently fortuitously, through great masses of anorthosite.

The St. Urbain ilmenites have been mined intermittently since 1908, those at Ivry since 1913. The total production to date from both places is probably in the neighbourhood of 30,000 tons, a little more than half of which appears to have come from one deposit at Ivry, the remainder from various occurrences in the vicinity of St. Urbain. Exact detailed figures as to output are not available, since it has not been customary to report the shipments from each property separately nor to always discriminate ilmenite in the published official returns. The ilmenite from St. Urbain runs from 35 to 40 per cent in TiO2 (21 to 24 per cent titanium), that from Ivry a trifle lower. The Ivry deposits, however, are the more favourably situated with respect to transportation. production from both localities has all been derived from surface workings, shallow hillside quarries worked entirely by hand.

Most of the ilmenite produced in Quebec-except the rutile-bearing ilmenite from the General Flectric Company's property—has gone to the Titanium Alloy Manufacturing Company of Niagara Falls, N.Y., to be used in the production of ferrotitanium, a purpose for which it has an economic advantage over the

more expensive rutile.

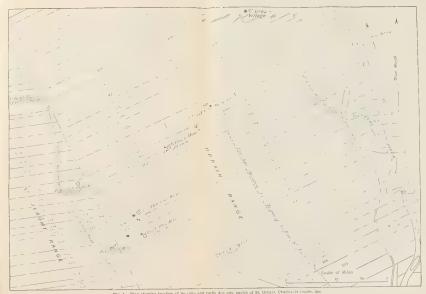
### PROVINCE OF QUEBEC

### Charlevoix County

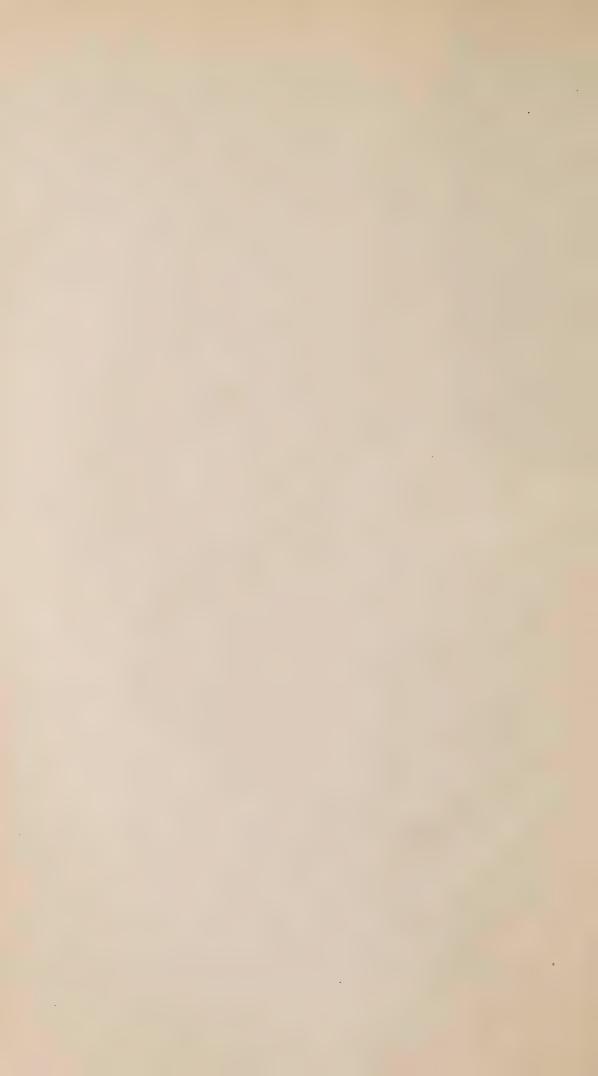
### PARISH OF ST. URBAIN

The ilmenite, or titanic iron, of St. Urbain is said to have been discovered in 1666 by Sieur de la Tesserie, and explorations were carried on in the following year by order of Colbert, Prime Minister of Louis XIV, King of France.1 The first serious attempt to utilize it, however, was made in 1871 or 1872, when the Canadian Titanic Iron Company commenced the erection of charcoal blast furnaces near the deposits, with the intention of making pig By November, 1873, two furnaces with the necessary appurtenances -blowing engines, charcoal kilns, etc.-had been completed and the furnaces blown in. They continued in blast during the following winter, making good white pig iron; but the output was only four or five, occasionally six tons per day, while the fuel consumption amounted, under the most favourable circumstances, to 190 to 237 bushels of charcoal (about 16 lb. per bush.) per ton of iron produced.2 Under such conditions the undertaking did not prove profitable, so after a few months of operation and the expenditure of about £80,000, the experiment was abandoned.3 All that now remains of this ambitious project is a few small heaps of slag and broken brick at the old furnace site. Small specimens of the pig iron produced at St. Urbain are still preserved in the museum of Laval University, in Quebec city; its composition has been given on a previous page (p. 27).

Wurtele, F. C., Historical record of the St. Maurice forges: Trans. Roy. Soc. Canada, vol. IV, 1886, p. 77.
 Harrington, B. J., Geol. Surv. Can., Report of Progress, 1873-74, pp. 249-251.
 Harrington, B. J., Trans. Amer. Inst. Min. Engrs., vol. 21, 1892-93, p. 865.



46410-p. 47



The possibility of utilizing the St. Urbain deposits again attracted attention when Rossi began to develop the manufacture of ferrotitanium from ilmenite in the electric furnace, and a small experimental shipment of the ore for this purpose was made some time previous to 1899. A further test shipment of 250 tons from the old ore piles left by the Canadian Titanic Iron Company in 1874 was made in 1908 to the Titanium Alloy Company of Niagara, Falls, N.Y. From 1908 to the present there has been an intermittent

small production from various properties in the district.

The productive ilmenite deposits of the parish of St. Urbain are situated along the southeasterly border of an undulating, thinly drift-covered plateau underlain by anorthosite that dominates the deep valley of the Gouffre river on the west. From Bouchard's workings-which are the most southerlynortheasterly through Coulomb's workings and the General Electric Company's workings to the old Furnace mine is nearly a mile (see Fig. 1). An early description picturing the deposits as an enormous bed 90 feet thick and continuous with minor interruptions for this distance is based on a former misconception of the geological structure of the district; the ilmenite does not form beds in a series of metamorphosed stratified rocks as was once thought, but occurs scattered in irregular bodies through massive igneous rock; and the various openings are not on a single continuous body but on a number of separate deposits. Some of the smaller bodies, in horizontal section, have the shape of flattened lenses, that over a small part of their length may have the appearance of veins or dikes, but which pinch out in a comparatively short distance at either end. It may be that the productive deposits form part of a mineralized zone in the anorthosite, but beyond the fact that some of the structural features of the different ore-bodies seem to have a tendency to orient hemselves in a common direction there is little to support such a supposition; ilmenite has been found at considerable distances on either side of such a possible zone, and the almost unbroken covering of surface soil, though thin, effectually conceals the frequency of its occurrence and its mode of distribution.

Although the development of none of the properties in the district has reached a stage that would warrant a quantitative estimate of the total tonnage available, there is ample evidence of the existence of very considerable and of ore in the Coulomb, the General Electric Company's, and the Furnace

nines alone.

The Glen prospect.—Some prospecting was done on an ilmenite deposit on lot 21 of the St. Urbain range, parish of St. Urbain, in 1911, by a Mr. Glen, of Montreal.

Two trenches at right angles to one another, dug through sand and clay in places eight feet deep, expose ilmenite, or titanic iron, for a distance of 35 feet from north to south and 30 feet from east to west. To the north and east the ilmenite body abuts against anorthosite; to the south and west it disappears under sand and clay. A pit sunk through clay 70 feet west of the trenches exposed anorthosite only. The depth of the overburden appears to have prevented further prospecting.

<sup>1</sup> Obalski, J., Report on the Mines of the Province of Quebec for 1899, Que. Dept. of Mines, 1900, p. 17.

The ore exposed is a pure massive ilmenite that yielded on analysis:—

SiO2		 					٠				•.•						,			٠	۰			٠	۰		1.68
FeO																											55.36
TiO2			٠	0		٠	۰									0 1		0	0	۰	٠	٠		٠	٠		38.29
S	 									٠	٠	٠	۰	٠		۰	٠	۰		۰	٠	٠	۰	۰	٠		0.041
P						۰								٠	c												Trace

Reference :-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que.

Bur. of Mines, 1912, pp. 83-85. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 114-116.

Bouchard's Mine.—On lot 622 of the St. Jérôme range, parish of St. Urbain, about a quarter of a mile east of the St. Jérôme range road and about half a mile from the Glen prospect, are the workings known as Bouchard's mine. Some 800 tons of titanic ore was shipped from here to the Titanium Alloy Company of Niagara Falls, N.Y., in 1910, but the narrowness and irregularity of the ore-body, as compared with neighbouring deposits, caused the working to be abandoned in the following year.

The ore was extracted from an open-cut, or rock trench, a little over 100 feet long, about 15 feet wide, and 15 feet deep in its deepest part, cut into a, hillside. As seen in the workings, the deposit takes the form of a branching vein or dike eight feet wide at the mouth of the open-cut. It narrows going westerly towards the face and splits, first into two and then into five or more tongues or branches. Two other tongues start out from the main trunk at the mouth of the cut and disappear, one to the north the other to the south, into its rock walls.

The country rock is a medium-grained grey anorthosite carrying in places a little disseminated titanic iron, the individual grains of which standing out on white weathered rock surfaces produce the effect of an indistinctly banded stippling. Black mica, also having a lineal arrangement that gives the rock a gneissoid appearance, occurs in places near the ore-body.

The ore is a dense, black, medium-grained ilmenite, showing little admixture of other minerals beyond a few scattered grains of pyrites. An analysis made for purposes of sale, quoted by Dulieux, showed it to contain 36.64 per cent titanic acid, equivalent to 21.98 per cent titanium. A general sample taken from the face in the open-cut and analysed in the Mines Branch laboratory by H. A. Leverin, yielded:-

<u>T</u> iO <sub>2</sub>	. 32.90
Fe	. 36.94
$\mathrm{SiO}_2$	. 1.68
S.,	. 1.46
Ti.,	. 19.74

Reference :-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec, 1911, Quebec Bur. of Mines, 1912, pp. 85-86. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp.

116-117.

Coulomb's Workings.—A short distance northeast of Bouchard's open-cut are the workings known as Coulomb's mine, on lot 319 of the cadastral plan of the St. Urbain range. A side road leading from the St. Jérôme range road

past Bouchard's working continues eastward to Coulomb's workings. In addition to lot 319, the property, which is now owned by the American Titanic Ore

Company of Quebec, includes adjoining lots 315, 318, and 320.

The deposits were first opened up in 1910 by a Mr. Coulomb, a local contractor, who during the following year took out some 1,500 tons of ilmenite and shipped it to the Titanium Alloy Company of Niagara Falls, N.Y. As is the case with all the other properties in the district, the work was done entirely by hand and the ore teamed to Baie St. Paul for shipment. Very little work has been done on the property since 1911.

The productive workings consist of two open-cuts, the first of which lies about 500 feet east of the line between the St. Jérôme and the St. Urbain ranges,

the second, 500 feet farther east. Both are on lot 319.

Western open-cut.—At the first, or western, open-cut, the surface soil has been stripped from the bed-rock, exposing ilmenite over an area about 90 feet from east to west and 75 feet from north to south. The exposed surface of the deposit slopes gently upward from west to east, and at right angles to the line of slope there has been blasted out across it a low working face, just behind which considerable broken ore is piled. On the south side of the stripping the ilmenite is in sharp contact with light-coloured massive anorthosite. Elsewhere the boundaries of the ore-body are hidden by overlying soil, so that the full extent of the occurrence could not be determined.

The ilmenite here is quite friable, at least to the shallow depth attained in the working, and readily breaks up into coarse powder composed of facetted grains of ilmenite. The chief impurities noticed were a little pyrite, partly in grains disseminated throughout the mass partly in thin parallel sheets that are rather plentiful in places, and light-coloured patches of earthy material resulting from the decomposition of embedded feldspars. The friability of the ore, which, though it facilitates breaking adds to the difficulty of handling without excessive loss of material, appears to be largely a surface characteristic hat will probably become less noticeable at greater depth.

A sample of about forty pounds of ore from the stock piles yielded on malvsis (Dulieux):—

$\begin{array}{cccccccccccccccccccccccccccccccccccc$	51.54 2.64 0.040
Iron	0 4 0 0

Eastern open-cut.—The second, or eastern, working is a comparatively marrow deep open-cut that follows an apparently dike-like body of ilmenite into the hillside; it is about 160 feet long and widens from about 20 feet at the mouth to 45 or 50 feet at the face, which is 20 or 25 feet high. Some surface stripping has been done and some ore taken out of two shallow pits just beyond the face. The continuation of the ore-body eastward, down the hill from the mouth of the open-cut, is concealed by drift.

The general impression gained from an examination of the workings is that of an irregular dike-like body of ore trending approximately east and west, the

46410 - 4

sides of the expavation corresponding roughly to the walls of the dike. On the north side of the open-cut the wall is fairly even and easily followed; it strikes nearly west, and the dip is to the south at an angle of about 80 degrees. The south wall is more irregular and uncertain; it shows at the mouth of the open-cut but disappears farther in.

The wall rock is a massive, medium-grained, light grey anorthosite composed almost entirely of feldspar. Except in places immediately adjacent to the ilmenite, where it may contain numerous plates of black mica oriented parallel

to the contact, it is comparatively free from dark minerals.

The ilmenite is less friable than that in the western open-cut and less pyrite is visible in it, but feldspathic bands and patches necessitate some sorting of the broken ore (the ore from the western workings is said to have been shipped without sorting). One such feldspathic band, about two feet wide, runs the length of the open-cut. It is made up of white porcelain-like feldspar, grains of titanic iron, and black mica, the dark minerals so arranged as to give the band a gneissoid structure parallel to the longer axis of the ore-body.

Analyses of the ore from the eastern open-cut, as given by Dulieux, are as

follows:-

	1	2
$TiO_2$	35.46	38.40
FeO	55.14	52.98
$\mathrm{SiO}_2$	3.12	2.68
P	0.044	0.041
S	0.040	0.040
Not determined	6.195	5.859
	100.00	100.00
Iron	42.89	41.21
Titanium	21.30	23.06

Numerous bore holes sunk to bed-rock through the over-burden of soil between the eastern and western open-cuts are said to have struck ilmenite, but there is no visible evidence that the two openings are on a single continuous deposit.

Reference:-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 86-91.
 Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 117-122.

The General Electric Company's Workings.—The General Electric Company's workings are between two and three hundred yards north of the Coulomb workings; on the eastern extremity of lot 324 of the cadastral plan of St. Urbain.

They have already been briefly described under the heading Rutile.

Possibily some of the ilmenite from this deposit was used in the Canadian Titanic Iron Company's furnaces during their brief campaign in the winter of 1873-74, since Harrington speaks of red grains of rutile in the ore treated, but the first work of any consequence was done on it in 1910, when the General Electric Company leased the property and for a short time worked it for rutile. Between 1911 and the present time the only shipments recorded were 1,400 tons in 1918.

In the various workings—surface strippings, pits, and open-cuts—included in an area about 300 feet long and 90 feet wide, considerable ilmenite is to be

seen, but whether it is in the form of a single compact mass or in a number of separate bodies is still a matter of conjecture.

Where free from rutile, the ilmenite is a dense, black, medium-grained mineral with a somewhat dull sub-metallic lustre, containing occasional grains and patches of feldspar and an odd speck of pyrites. Warren finds from a microrespical study of polished surfaces that it is not homogeneous but is made up fine interlamellar intergrowths of ilmenite and hematite. Its chemical com-

position is indicated in the following analysis (Dulieux):-

the state of the s	
$TiO_2$	41-61
FeO	57.24
$\operatorname{SiO}_2$	1.10
P	Trace
S	"
Undetermined	0.06
	10001
	100.01
Iron	44.52
Titanium	24.98
	- 1 00

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 91-94. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp.

122-125.

Warren, C. H., The ilmenite rocks near St. Urbain, Quebec: Amer. Jour. Sci., 4th Series, vol. 33, 1912, pp. 263-277. Abstracted by T. L. Watson in U.S. Geol. Sur. Bull. 580, 1915, pp. 404-408

Harrington, B. J. (Discussion), Trans. Amer. Inst. Min. Engrs., vol. 21, 1892-93,

Furnace Mine.—Northeast of the General Electric Company's workings there are no known exposures of ilmenite in the prevailingly drift-covered stretch of three-eighths of a mile between them and the Furnace mine. This latter is located on the line between lots 361 and 352 as shown on the cadastral plan of the parish of St. Urbain and gets its name from having been the scene of operations of the Canadian Titanic Iron Company, whose furnaces stood on lot 364, some distance below the openings from which they drew their ore. Since the failure of the company in 1874, nothing appears to have been done on the deposit, and all that can be seen at the old mine workings is two partly earthfilled open-cuts about 120 feet apart in a hillside with, halfway between them, the remnants of a couple of charcoal kilns or roasting stalls.

In one of the open-cuts, ilmenite, with some included anorthosite, is still exposed over a width of 60 or 70 feet; the other excavation, which is cut in two by an old road, is completely filled with earth on the downhill side, below the word, but on the uphill side there is still exposed an elliptical quarry face about 100 feet long, practically all in ilmenite. The dilapidated condition of the workings and the surrounding covering of surface soil effectually conceal any further evidence of the extent of the ore-bodies. Obalski, who visited the spot in 1898, mentions three openings: first, a "breast of solid ore 100 by 100 feet with a height of 20 feet"; second, a face 30 feet high and 60 feet wide; third, a cutting on the ore-body 100 feet long, 50 feet wide, and 50 feet high.

The surrounding rock is anorthosite, much of it having a green or pink tinge but in parts pure white and marblelike. The ore is the usual dense, black, medium-grained ilmenite and appears to be remarkably pure except for small grains of pyrite rather uniformly scattered through it.

 $46410 - 4\frac{1}{2}$ 

Two analyses of the ore from the Furnace mine, made in the Mines Branch laboratory by H. A. Leverin, are as follows:-

	1	2
Γί. Fe. SiO <sub>2</sub> . P. S. V <sub>2</sub> O <sub>5</sub> . Cr <sub>2</sub> O <sub>3</sub> . Ni		0.058 0.148 0.09 0.25 Nil

References:

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 94-96. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp.

Harrington, B. J., Geol. Sur. Can., Rep. of Progress for 1873-74, pp. 249-250. Obalski, J., Rept. on Mines of the Province of Quebec, Dept. of Colonization and Mines, Quebec, 1900, p. 17.

Bignell Prospect.—Ilmenite has been found in several small test-pits dug through the clay overburden a short distance east of the St. Jérôme range road, on lot 608 of the St. Jérôme range, parish of St. Urbain. This occurrence is about half a mile west of a line drawn through the productive workings already described. (See sketch map, Fig. 1.)

Most of the pits have now fallen in, but ilmenite in place is visible in some of them, and pieces of the broken ore still lie alongside others. The exposures, however, are altogether insufficient to convey any information as to the extent of the deposit. The work is said to have been done by the Westinghouse Electric Company.

To ascertain whether or not there was a continuation of the deposit, Dulieux, in 1911; had three test pits dug on the adjoining lot, 609, at points where the dip needle showed local attraction. Two of these holes reached anorthosite only, the third was abandoned after passing through eight feet of sand and clay.

It may here be said that the magnetometer and dip needle, useful instruments for locating hidden deposits of magnetic iron ore, are of no service in a search for titanic iron in this district, for the ilmenite masses have little or no appreciable effect on the magnetic needle. The magnetic variations found in the neighbourhood are to be ascribed to the influence of small quantities of magnetite that are found at places disseminated through certain phases of the country rock, not to ilmenite.

Reference :-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 78, 96. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, p. 127.

Lot 641, St. Thomas Range.—About a quarter mile west of the St. Thomas Range road (about two miles and a quarter west of the Bignell prospect) there is a mineralized outcrop, 6 feet by 4 feet, showing impure titanic iron. The occurrence is on lot 641 of the St. Thomas range, parish of St. Urbain, close to the line between lots 640 and 641.

The enclosing rock is a friable anorthosite and the ilmenite is intermixed with much feldspar.

Décharge Range.—A little prospecting on the east bank of the river Gouffre, about three miles above the village of St. Urbain, on a lot in the Décharge range, has exposed a band six or seven feet wide of mixed ilmenite, feldspar, and ferromagnesian minerals. The wall-rock is massive grey anorthosite. The band disappears under the surface soil at either end.

### QUEBEC SEMINARY LANDS

Ilmenite has also been found 18 or 19 miles by wagon road north of St. Urbain, on land belonging to the Quebec Seminary, in the seigniory of Beaupré. The occurrence is about four miles and a half northwesterly from Pitre Tremblay's mill on the Gouffre river, along a wagon road leading to the old post road to Chicoutimi. The exposure, about five feet in diameter, is bounded on one side by grey anorthosite; elsewhere it disappears under drift. No work has been done to show the extent of the deposit.

At the surface the ilmenite has disintegrated into ochre-covered grains having curved faces with polygonal outlines; but it becomes solid at a depth of a few inches.

Analyses of the granular surface material (1) and of solid ore from a point six inches below the surface (2) are as follows (Dulieux):-

	1	2
TiO <sub>2</sub> . F <sub>0</sub> O. SiO <sub>2</sub> . P. S. Undetermined.	$31 \cdot 28$ $65 \cdot 16$ $2 \cdot 50$ Trace $0 \cdot 040$ $1 \cdot 02$	$\begin{array}{c c} 32 \cdot 25 \\ 63 \cdot 22 \\ 2 \cdot 00 \\ \text{Trace} \\ 0 \cdot 042 \\ 2 \cdot 488 \end{array}$
Iron Titanium	$100 \cdot 00$ $50 \cdot 68$ $18 \cdot 78$	$ \begin{array}{c c} 100.00 \\ 49.17 \\ 19.37 \end{array} $

### Reference :-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 97-98. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp.

128-129.

### Terrebonne County

### BERESFORD TOWNSHIP

Outside the parish of St. Urbain, in Charlevoix county, the only locality at which ilmenite has been worked in Canada is near Ivry, in Beresford township, Terrebone county, Quebec. The ore here also occurs in anorthosite under geological conditions similar in all respects to those obtaining at St. Urbain.

Ivry Mine.—The workings known as the Ivry mine are on lot 38 and lot 37 west in the fifth range of Beresford township, two and one-half miles from Ivry, a station on the Montreal-Mont Laurier branch of the Canadian Pacific railway, 67 miles north of Montreal. The nearest village of any considerable size is Ste. Agathe on the same railway, from which the workings can be reached by five miles of good wagon road.

The country in the vicinity of Ivry consists of steep rounded hills separated by drift-filled valleys, or sunken plains, watered by numerous lakes and streams. The ilmenite deposits, which lie some 300 yards south of the road, are exposed in an open-cut and a number of pits and trenches that have been excavated part way up the northeast face of one of these hills that rises some 300 feet above Manitou (Morin) lake to the south. Bed-rock in the vicinity is covered with drift, and the only exposures are those to be seen in the workings. These are mostly on lot 38 and are spread over an area about 1,100 feet long by 120 feet wide, the trend of the longer dimension being about WNW. (See Fig. 2.)

The largest exposure, an open-cut from which ore has been extracted for shipment, is in the form of a rough quarry face about 100 feet wide and perhaps 30 feet high, blasted out of the steep hillside. The face is practically all in ore. In their present condition the smaller prospect openings afford very little definite information beyond the fact that ilmenite occurs at a number of places, probably as individual pockets, or masses, distributed through the enclosing anorthosite, rather than as a single continuous ore-body. The deposit is non-magnetic so the dip needle could not be used to determine this point definitely.

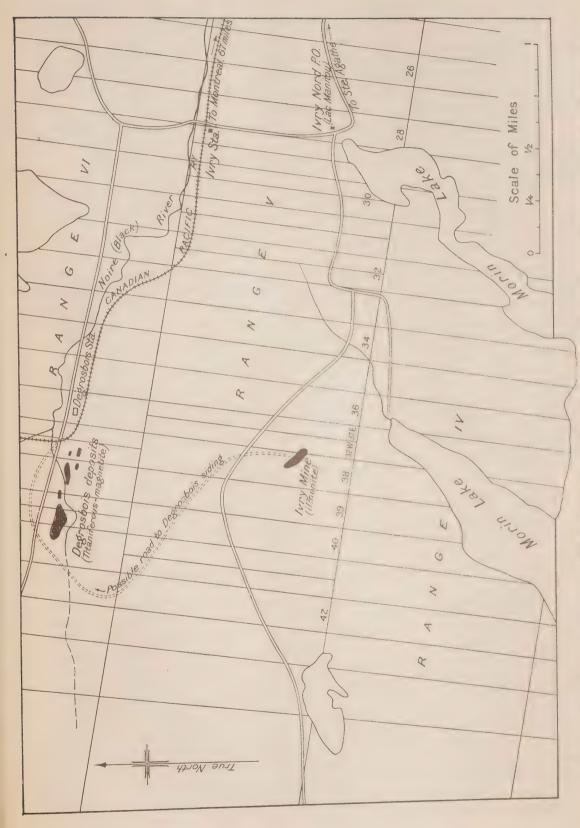
The ore is a hard, medium to coarse-grained ilmenite. Except that occasional crystals of feldspar and scattered grains of pyrite are distributed through the mass, particularly near its borders, it is very free from intermixed material.

The country rock is massive anorthosite, dark purple in colour and without signs of foliation. In addition to feldspar, it contains, at places, a little pyroxene, disseminated grains of titanic iron, and flakes of black mica. In small joint planes near its contact with the ilmenite there is also some pyrite. The transition from ore to wall-rock is abrupt, and there is no trace of crushing or movement near their junction.

The chemical composition of the ore is indicated in the following analyses, of which the first two are taken from Dulieux. The third is of a general sample from the quarry face analysed in the Mines Branch laboratory by H. A. Leverin, and the last, a special sample, also analysed in the Mines Branch laboratory, was selected with a view to determining the presence of some of the rarer elements:—

	1	2	3	4
Ti Fe SiO <sub>2</sub> P S V <sub>2</sub> O <sub>3</sub> CroO <sub></sub>	48.05			$   \begin{array}{c}     19.84 \\     42.98 \\     \hline     0.076 \\     0.144 \\     0.04 \\     \hline     0.02 \\     \hline     0.03 \\     0.04 \\  $
Cr <sub>2</sub> O <sub>3</sub> Ni				0.08 Nil.

The Ivry deposits are well located, both for cheap mining and with regard to transportation facilities, and have in this last respect a distinct advantage over those at St. Urbain. The workings are at such an elevation that the ore could be delivered to the railway at Degrosbois siding two miles away by aerial tram, or a wagon road having a down grade all the way could be laid out to the same shipping point.



Plan showing location of the Ivry (illnenite) mine and Degrosbois (titaniferous magnetite) deposits, Beresford township, Terrebonne county, Que. 31 Fig.

The first shipment of ilmenite from the Ivry mine was made in 1912, and there has been a small, intermittent production since that time, the last shipments having been recorded in 1918. The total production to date is probably in the neighbourhood of 16,000 tons (the exact figures are not available), all of which was sent to the Titanium Alloy Company of Niagara Falls, N.Y., to be used in the manufacture of ferrotitanium. All the ore was mined by hand and hauled by teams to Ivry or Degrosbois siding for shipment.

Though the development work done at Ivry is not such as to afford sufficient information on which to base tonnage estimates, it discloses nothing that would lead to a belief that the total quantity of ilmenite available is not large.

Reference:-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 66-71. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 131-135.

### MINOR OCCURRENCES OF ILMENITE

Occurrences of ilmenite, or titanic iron, associated with anorthosite have been noted by Adams in other portions of the great Morin mass in which the Ivry deposits are found, and by Low, at many places in the great anorthosite masses lying north of the river and gulf of St. Lawrence. These occurrences, though of no present economic interest, serve to illustrate the widespread association of ilmenite with the anorthosite rocks of Quebec.

A certain amount of ilmenite is also found in the magnetic black sands along the north shore of the river and gulf of St. Lawrence, but in such small amount that except in the presently improbable contingency of these being worked for iron and the ilmenite recovered as a by-product of their concentration, they are not likely to serve as a source of supply of titanium.

Adams, F. D., Geol. Sur. Can., Annual Report, vol. 8, 1895, pp. 100-101 J, 141 J. Low, A. P., Geol. Sur. Can., Annual Report, vol. 8, 1895, pp. 202 L, 229 L, 232 L, 287 L.

Dulieux, P. E., Les Minerais de fer de la Province de Québec, Que. Bur. of Mines.

1915, pp. 137-164. Mackenzie, G. C., The Magnetic Iron Sands of Natashkwan, Saguenay County, Quebec, Mines Branch, Dept. of Mines, Pub. 145, Ottawa, 1912.

### TITANIFEROUS MAGNETITE OCCURRENCES

Titaniferous magnetite is of more frequent and more widespread occurrence in Canada than ilmenite, but known deposits of workable size are, with one possible exception, confined to the provinces of Ontario and Quebec. Many of the Quebec occurrences are associated with the same anorthosite masses in which the ilmenite deposits are found, usually in portions of the anorthosite richer in ferromagnesian minerals and consequently more nearly approaching ordinary gabbro in composition. Practically all the Ontario titaniferous magnetites (if magnetite containing less than 2 or 3 per cent titanium is excluded from this category) are found in gabbro, as ultra-basic segregations in phases of the gabbro so rich in ferromagnesian silicates as to approach pyroxenite in appearance. Unlike the ilmenite ore-bodies, which commonly show a fairly sharp line of separation from the enclosing rock, the deposits in gabbro usually pass from ore to rock by an almost imperceptible gradation, and their boundaries are more or less indefinite.

Concerning the origin and mode of occurrence of titaniferous magnetites in general, J. F. Kemp says<sup>1</sup>:—

serve to distinguish them.

A moment's reflection will make it evident that this tendency to vary, or, in other words, the tendency of some minerals to aggregate to the exclusion of others, may lead to the production of important bodies of iron ore. If a normal gabbro containing labradorite, pyroxene, olivine, and magnetite in the proportions of say 3:3:1:1, becomes gradually enriched with magnetite almost to the exclusion of the other components, a change takes place that is entirely analogous to the familiar passage of a normal gabbro into a pyroxenite or an anorthosite, but it is one that is less frequent and of a lower order of magnitude, because magnetite is a less abundant component than is either the pyroxene or feldspar. The development of bodies of magnetite in this way is nevertheless well known in several parts of the world............. The extremely basic portions of the magma, which have yielded the large bodies of ore, have been developed while the rock mass was still, at least in large part, a molten fluid, and the rearrangements have been essentially magmatic. The chemical and physical laws which underlie the rearrangements are obscure, and as time goes by artificial experiment may be needed to elucidate and demonstrate them.

Canadian titaniferous magnetites seldom carry more than 20 or 25 per cent TiO<sub>2</sub>, equivalent to 12 or 15 per cent titanium, and for this reason are unable to compete commercially with the richer ilmenites as titanium ores. On the other hand, they are not desirable as iron ores, first, on account of their titanium content, second, because they do not carry enough iron. Analyses of Canadian titaniferous magnetites seldom yield 50 per cent or over of iron, and it is extremely doubtful whether any of the known deposits are capable of maintaining any considerable continuous production running over 40 or 45 per cent, without resorting to hand sorting or concentration of some kind. They have at present no economic use.

The most extensive of the Canadian titaniferous magnetite deposits are those near Seine bay, in western Ontario. Next in point of size are probably those found in Bourget township, in the Lake St. John district, Quebec. Seemingly extensive deposits also occur at Bay of Seven Islands, on the lower St. Lawrence, Quebec. Over certain areas in central Ontario underlain by gabbro, small deposits of titaniferous magnetite are of frequent occurrence, but individual bodies of large size are not known. Since there is no demand for the material contained in them, very little work of any kind has been done on any

of the deposits.

<sup>&</sup>lt;sup>1</sup> Titaniferous iron ores of the Adirondacks; U.S. Geol. Sur., 19th Annual Report, 1897-98, pp. 383-384.

### PROVINCE OF QUEBEC

### Chicoutimi County

### BOURGET TOWNSHIP

St. Charles Prospect.—Outcrops of titaniferous magnetite occur on the left bank of the Saguenay river, on lot 44 in range I of Bourget township, about a mile and a half west of the little village of St. Charles. They can be reached either by leaving the Quebec and Lake St. John branch of the Canadian National railway at Jonquière and driving the intervening 17 miles—the Saguenay river being crossed by ferry at St. Charles village—or by leaving the same railway at Hébertville and driving by way of the Alma bridge and Archambault road, a distance of 24 miles, to the farm house of Jean Brossard, on lot 43, close to which the deposits are located.

The country rock in the vicinity of the deposits is a foliated variety of gabbroic anorthosite, part of the great anorthosite mass that extends many

miles eastward from lake St. John.

The most accessible, and largest, outcrops of titaniferous magnetite lie within a few hundred feet of the Saguenay, where they constitute much of the exposed bed-rock on a hillock, or spur, that juts out like a promontory from the line of wooded heights that dominate the river. Numerous outcrops can be seen both at the foot and on top of the hillock, scattered over an area about 700 feet long and at least 200 feet wide. The largest seemingly continuous outcropping of magnetite occupies a lozenge-shaped area on top of the hillock and is about 320 by 200 feet in its greater dimensions. It is not all magnetite, however, there being some included tongues and masses of anorthosite. Immediately north of the lozenge-shaped area titaniferous magnetite outcrops are also numerous over a second triangular area of about 10,000 square feet. Still farther north are some small lenses of magnetite entirely surrounded by anorthosite. To the northeast the outcrops show only anorthosite.

Six or seven hundred feet northwesterly from the outcrops near the river, on what may be described as a sort of wooded plateau, numerous outcrops, all showing titaniferous magnetite, are again found over an area 300 feet long in a southwesterly direction, and having a maximum width of 200 feet. A similar area of about the same size occurs some distance still farther on, in the same general direction, but its exact location was not determined.

Excepting one or two shallow holes in the outcrops near the river, no work whatever has been done on the deposits, and the country inland is entirely unprospected.

The ore-bodies at the river are admirably situated for quarrying operations and, in a straight line across the river, are only four miles from the railway. Should it at any time be desired to treat the ore electrically, large amounts of hydro-electric power have been developed within 15 or 16 miles of the deposits.

Analyses of the St. Charles ore, made in the Mines Branch laboratory by H. A. Leverin, are as follows:—

Ti	12.60
Fe 50.50	52.10
$SiO_2$	
P 0.046	0.079
S 0·492	0.034
$V_2O_5$	0.10
$\operatorname{Cr}_2\operatorname{O}_3$	0.07

Some laboratory tests on magnetic concentration of the St. Charles ore were made by Dulieux, with the following results:-

50.53% 10.55%

This was divided by screening into three sizes, A, B, and C, and each size was treated separately.

		Crude	cre		Con	centra	tes	Tailings			
Size		Anal	ysis		Per   cent	Anal	ysis	Per cent	Analysis		
	Fe	Ti	S	Р	ore	Fe	Ti	ore	Fe	Ti	
Size A: from 20 to 40 mesh, 39·40 per cent of sample		12·01 11·36	0·020 0·018 0·012 Loss 0	0.026 $0.050$		57·50 59·68 50·02	9.39	25·80 23·00 17·40	31.00	21.59	

By re-calculating the above figures we obtain for the unsized ore:—

	Crude	Concentrates (77 per cent of crude)	Tailings (23 per cent of crude)
Iron Titanium Titanium dioxide (TiO <sub>2</sub> ) Sulphur Phosphorus.	50·53 10·55 17·6 0·63 0·03	56·2 8·3 13·8	20·9 19·7 32·8

### References—

Dulieux, P.E., Report on Mining Operations in the Province of Quebec, for 1912, Quebec Bureau

of Mines, 1913, pp. 84-90.

Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 52-58.

Stansfield, Alfred, Electric smelting as a means of utilizing the iron ore of the St. Charles deposit; Geol. Sur. Can., Memoir 92, chap. 6, 1916, pp. 52-73.

### KENOGAMI TOWNSHIP

A little prospecting has been done on some occurrences of titaniferous magnetite on range II, Kenogami township, Chicoutimi county, near Samson station on the Quebec and Lake St. John branch of the Canadian National railway. The excavations lie north of the track between mile posts 208 and 209. The country in the immediate vicinity is hilly, barren, and desolate. The nearest village of any importance is Jonquière, nine or ten miles away.

All that can be seen in the few shallow pits and trenches is a little titaniferous magnetite mixed with much pyroxene and feldspar. Apparently, the deposits constitute small segregations in the surrounding granular pyroxenebearing anorthosite. They are too small to be of economic significance.

A selected sample of the magnetite yielded (Dulieux):-

Some ten carloads of the ore are reported to have been shipped to the blast furnace at Radnor, in 1901.

### Reference:-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1912, Que.

Bur. of Mines, 1913, pp. 91-93.

Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 58-60.

Obalski, J., Report on Mining Operations in the Province of Quebec for 1901, p. 10.

### Lake St. John County

### ILE D'ALMA

Alma island lies in the Saguenay river at the outlet of lake St. John, where it divides the river into two branches, the Grande Décharge and the Petite Décharge. On lot 36, range II of the canton de l'Ile, about 800 feet north of the road which crosses the island from east to west, a series of small lenses of hard compact titaniferous magnetite occur in coarse-grained anorthosite. The largest lens exposed in the outcrops measures 15 by 30 feet. The deposits are too small to be of any importance.

A sample of the ore yielded on analysis (Dulieux):—

| Iron     | <br> | 53.07 |
|----------|------|------|------|------|------|------|------|------|-------|
| Titanium | <br> | 11.94 |

### Reference:-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1912, Que. Bur. of Mines, 1913, p. 93.

Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 60-61.

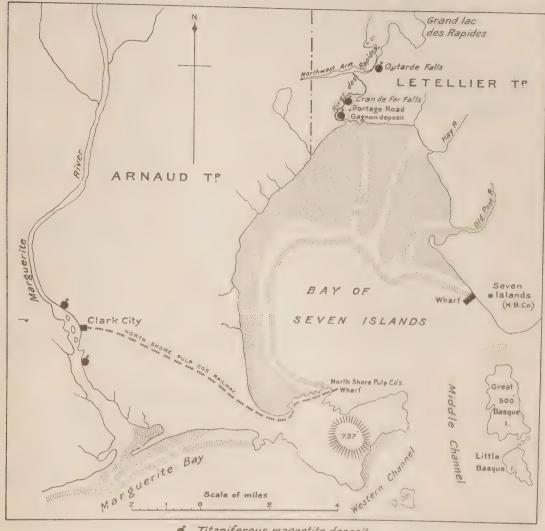
### Saguenay County

### BAY OF SEVEN ISLANDS

Bay of Seven Islands, 340 miles below Quebec city, is the most important centre of activity on the north shore of the lower St. Lawrence river. The village of Seven Islands, on the east side of the bay, is the seat of a Roman Catholic bishopric and of fur-trading posts of the Hudson Bay Company and Révillon Frères. On the west side is the wharf of the North Shore Pulp Company, which is connected by 9 miles of railway with the pulp mills and hydroelectric power plant of the same company at Clarke city, on the Marguerite river. During the summer months a freight and passenger steamer plies fortnightly between Quebec city and Bay of Seven Islands.

Titaniferous magnetite deposits are found in the vicinity of Bay of Seven Islands both on the river Des Rapides, which empties into the end of the bay, and on the Marguerite river, near Clarke city, west of the bay. (See Fig. 3.) The more important deposits are those on the river Des Rapides, north of the bay, and these are the ones usually referred to in various government reports as the Seven Islands deposits. They are mentioned by T. Sterry Hunt, of the Geological Survey of Canada, as early as 1869 and subsequently by J. Obalski

in a number of the reports of the Quebec Department of Colonization, Mines and Fisheries. Between 1867 and 1875, when the Moisie Iron Company was operating, titaniferous magnetite was brought from Cran de Fer falls on the



Titaniferous-magnetite deposit

Fig. 3. Sketch map showing location of titaniferous magnetite deposits, Bay of Seven Islands, Saguenay county, Que.

river Des Rapides to be mixed with the black sand used in the bloomeries at Moisie.

Obalski, in the Quebec report for 1903, gives the results of an attempt to treat the river Des Rapides ore on a Wetherill magnetite separator.

### RIVIERE DES RAPIDES

Between the mouth of the river Des Rapides and the outlet of Lac des Rapides, a distance of about four miles as the river flows, the rock formation consists of gabbro and anorthosite, these two varieties of igneous rock alternating at places in light and dark-coloured bands. The anorthosite proper carries only

a few disseminated grains of titaniferous magnetite but the gabbro is almost everywhere highly charged with it; and at two points at least, Cran de Fer falls and Outarde falls, titaniferous magnetite becomes so abundant, to the exclusion of other minerals, as to form distinct, well-defined deposits. Of the three varieties of rock to be seen in juxtaposition at these places—anorthosite, gabbro, and titaniferous magnetite—the boundaries between the first and either of the last two are always sharp and well-defined; but the transition from gabbro to titaniferous magnetite is frequently brought about by a gradual increase of iron-titanium constituents in the gabbro. Sometimes, however, granular grey gabbro and compact black magnetite lie in adjoining, but distinct, parallel bands.

Cran de Fer Falls: Molson Mine.—At the second fall above the mouth of the river Des Rapides—Cran de Fer falls—are the shallow surface workings known as the Molson mine, from which ore was extracted during the period 1867-1875. The old workings on the river bank are small and insignificant.

Much of the rock exposed on both banks of the river at the falls and for a considerable distance above is a magnetite-rich gabbro that grades locally into substantially pure titaniferous magnetite. The total quantity of magnetite available at this place is apparently large, but the irregularity of the occurrence, the wide variations in its composition, and the difficulty of distinguishing ore from some of the darker phases of the gabbro, make a definite estimate of the quantity at present impossible.

Of the following analyses, taken from Dulieux, the first four were made on samples of magnetite-rich gabbro, the last four on samples of titaniferous

magnetite:-

### Analyses of Magnetite-rich Gabbro from Rivière Des Rapides

	Ti	Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	S	Р
1 2 3 4	$0.225 \\ 0.71 \\ 4.51 \\ 5.60$	$28 \cdot 60$ $22 \cdot 06$ $21 \cdot 82$ $16 \cdot 15$	14.26	$\begin{array}{c} 20 \cdot 00 \\ 11 \cdot 67 \end{array}$	9.48	2·16 1·28	0.21	1·2 1·68

### Analyses of Titaniferous Magnetite from Rivière Des Rapides

	Ti	Fe	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	S	Р
5 6	14.73 $13.46$ $12.85$	45.70 $52.84$	6.72				0.13	0.0
8	15.51	51.85	1.16	1.50	0.32	3.42	• • • • • • • •	

A sample weighing 3,000 pounds was taken from an ore pile that had been left at the old workings and sent to the Mines Branch testing laboratory for treatment by magnetic separation, with the following results:—

The ore was ground in a ball mill until 54.65 per cent would go through

a 200-mesh sieve then passed through Gröndal separators, yielding:-

·	Crude ore	First concen- trate (52.9 per cent of crude ore)	First tailing (47·1 per cent of crude ore)
Fe (soluble). TiO <sub>2</sub> . SiO <sub>2</sub> . P. S.	$\begin{array}{c} 41 \cdot 2 \\ 25 \cdot 6 \\ 9 \cdot 36 \\ 0 \cdot 21 \\ 0 \cdot 71 \end{array}$	55.28 $19.8$ $0.66$ $0.02$ $0.443$	28·01 35·26

The concentrate thus obtained was again ground in the ball mill and passed through the separators, with the following result:—

	Second concentrate (86·2 per cent of first concentrate)	(13.8 per cent of
Fe	57·35 16·9 0·2 0·010 0·390	40·1 48·34

The second concentrate was ground in the ball mill for the third time, until 73.7 per cent would go through a 200-mesh sieve and then passed through the Gröndal separators for a third time, yielding:—

	Third concentrate (38.16 per cent of second concentrate)	Third tailing (61.84 per cent of second concentrate)
Fe	58·75 14·85	55·3 16·33

The redistribution of iron and titanium as effected by the three separations is:—

	Per cent of total	Per cent of total
In final concentrate	25.17	$9.43 \\ 90.56$

These results would indicate that it is not feasible to obtain a substantially titanium-free concentrate from this ore; but a very pure ilmenite product can be made by magnetic separation.

References :-

Dulieux, P. E,. Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 105-114. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp.

61-72.

Mackenzie, G. C., Experiments in magnetic separation with a titaniferous magnetite from Rivière des Rapides, Mines Branch Summary Rept. for 1912, pp. 61-67.

Outarde Falls Deposits.—Outarde falls are about one and one-half mile above the Cran de Fer falls; they are the fourth falls met in ascending the river Des Rapides from its mouth.

The rock at Outarde falls is a foliated gabbro-anorthosite. Above the falls it is light grey in colour and coarse-grained, but towards the foot of the falls it changes, becoming granular and progressively richer in magnetite, while at the same time the foliation becomes more accentuated. On the right bank of the river, about 100 feet below the falls, the gabbro, in places, is so heavily charged with magnetite as to be really a titaniferous iron ore. In an outcrop extending about 60 feet along the right bank of the river all gradations from gabbro to practically pure magnetite can be seen. Solid magnetite, however, constitutes but a small proportion of the whole. A little surface work has been done in this vicinity.

On the left bank the only exposure is a small outcrop of titaniferous magnetite about 100 feet from the river.

Analyses of the magnetite-rich gabbro on the right bank (1) and of a sample from the outcrop on the left bank (2) are as follows (Dulieux):-

-	. 1	. 2
Iron. Titanium Sulphur. Phosphorus.	$25.75 \\ 10.53$	$54 \cdot 99$ $10 \cdot 88$ $0 \cdot 08$ $0 \cdot 033$

Gagnon Deposit.—A large area on the left bank of the river Des Rapides between its mouth and the first falls is underlain by a dark gabbro, in some places so charged with grains of titaniferous magnetite as to have the appearance of solid ore. Good exposures of this material can be seen in the vicinity

Analyses of samples taken from two different outcrops yielded (Dulieux):—

	1	2
FeO	33 · 18 18 · 62 11 · 78 8 · 29	36.48 $14.62$ $12.08$ $1.61$
Iron Titanium	25·81 11·18	28·37 8·79

Reference:-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que.

Bur. of Mines, 1912, pp. 114-116. Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 71-73.

### MARGUERITE RIVER

Clarke City Deposits.—The village of Clarke City, together with the mill and power plant of the North Shore Pulp Company, is situated on the Marguerite river, about four miles due west of the west shore of Seven Islands bay.

On the left bank of the river, half a mile below the company's dam, gabbro-anorthosite carrying much disseminated magnetite is exposed over an area about 400 feet long and running back 50 to 60 feet from the water's edge. Scattered over a portion of this area are several lenticular masses of fairly pure titaniferous magnetite, the largest of which is 80 feet long and 15 feet wide.

Analyses of two samples taken from these lenticular masses yielded (Dulieux):—

	1 .	2
FeO	$ \begin{array}{c c} 71.38 \\ 20.68 \\ 1.52 \\ 0.049 \\ 0.23 \end{array} $	$68 \cdot 18$ $16 \cdot 40$ $7 \cdot 88$ $0 \cdot 013$ $0 \cdot 59$
Corresponding to: Iron Titanium	$\begin{array}{c} 55\cdot 10 \\ 12\cdot 42 \end{array}$	53.03 $9.84$

As the bed of the Marguerite river is here cut through terraces of sand and clay, the only other mineralized outcrop of bed-rock to be seen in the vicinity of Clarke City is in a quarry on the left bank of the river, at the water's edge, about half a mile above the falls. The quarry is an elliptical opening 165 feet long by 65 feet wide, from which stone was taken to build the pulp-mill and dam. The quarry floor is cumbered with debris so that the rock in place is visible only in the walls. These consist of rather coarse-grained anorthosite carrying in places much magnetite in the form of grains and masses.

A general sample was taken from an area of about 70 by 30 feet on the north side of the quarry (1), and a second sample of a small band about 3 feet wide of particularly pure ore included in the larger area (2); both were analysed, with the following results (Dulieux):—

	1	2
Fe Ti SiO <sub>2</sub> CaO MgO P S	38.86 9.06 15.96 3.87 3.57 0.08 0.187	57·84 11·34 0·84 0·013 0·51

The extent of the mineralized area beyond the quarry walls is concealed by the overburden of sand and clay.

<sup>46410-5</sup> 

### CHALOUPE RIVER DEPOSITS (CAP ROND)

The Chaloupe river empties into the gulf of St. Lawrence about 60 miles

east of Seven Islands bay.

Between the mouth of the river and Cap Rond, nearly three miles farther east, the rocky shore consists of anorthosite, usually foliated and containing, parallel with the foliation, numerous darker discontinuous bands that are heavily charged with titaniferous magnetite. The magnetite-rich portions of the anorthosite are of no great size, and only in spots do they carry as much as 60 per cent magnetite, the other constituent being feldspar identical with that of the surrounding anorthosite.

Two samples from Cap Rond, consisting of titaniferous magnetite, feldspar,

pyroxene, and some sulphides, yielded on analysis (Dulieux):-

Between the Chaloupe river and a point two or three miles east of Cap Rond, none of the deposits in the immediate vicinity of the sea shore are of any economic importance.

### THUNDER RIVER (RIVIERE AU TONNERRE)

The little settlement at the mouth of Thunder river is on the north shore of the gulf of St. Lawrence about 13 miles east of Chaloupe river.

On a hill about a mile and three-quarters inland, northeast of the settlement, there is an outcrop of bed-rock showing titaniferous magnetite surrounded by white-weathering anorthosite. The exposure of titaniferous magnetite is pear-shaped, about 50 feet long and 35 feet wide at its widest part. It is sharply outlined against the enclosing anorthosite, there being no zone of transition from ore to wall-rock.

An analysis of the ore, which is hard and only slightly magnetic, yielded (Dulieux):—

It should probably be classified as ilmenite.

The outcropping is the only one to be seen in the vicinity. Reference:—

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1911, Que. Bur. of Mines, 1912, pp. 119-133.

Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 75-88.

### St. Maurice County

### SHAWINIGAN TOWNSHIP

Grondin, or Shawinigan Mine.—St. Boniface de Shawinigan is a village situated on the Canadian National railway, about half way between Montreal and Quebec. It is 82 miles from Montreal and 92 miles from Quebec. About four miles by good wagon road from the village, the titaniferous magnetite occurrences known as the Grondin mine are found, on lots 22 and 23, range VII, in Shawinigan township.

In 1878, a small charcoal blast furnace was built on the bank of the Yamachiche river, on lot 17, by one Grondin, and an attempt was made to smelt the ore from the deposit on lot 22. Titaniferous magnetite, limestone, and

charcoal were first charged, but after four days the furnace became blocked. It was cleaned out, repaired, and again blown in, this time on bog ore from St. Paulin mixed with a little of the titaniferous ore. It was then found possible to make good pig iron, which was shipped to Three Rivers, but the enterprise was abandoned after about two months' operation.

The surrounding country is, for the most part, flat, but is broken here and there by low rocky hills protruding through the sandy soil. It is in a series of such hills, some of which rise 100 feet above the general level of the plain, that the Grondin mine is situated. The country rock in the vicinity of the deposits is a ferromagnesian rich gabbro composed of feldspar, pyroxene, and

titaniferous magnetite.

The greater portion of both lots 22 and 23 are wooded, and outcrops of bed-rock are few. On lot 22 the only excavation is the Grondin mine, a trench 22 feet long, 10 feet wide, and 6 feet deep, from which was obtained the ore used in the smelting operations carried on over forty years ago. The trench is all in ore of low grade. East of the trench several outcrops show the presence of titaniferous magnetite under a thin covering of soil, the largest exposure being along a small rocky ridge 10 feet high and 50 feet wide. High dip needle readings were obtained over an elliptical area 175 feet long by about 60 feet wide, in the vicinity of the trench.

The showings on lot 23 are unimportant. One is a low rocky mound consisting partly of massive gabbro and partly of basic, foliated gabbro containing some streaks of titaniferous magnetite; over a width of 25 feet magnetite constitutes perhaps 40 or 50 per cent of the rock; one band 18 inches wide is over 75 per cent magnetite. The other exposure is in an excavation 15 by 15 by 4 feet, in a meadow adjoining the road, and consists of a band, or lens, of titaniferous magnetite only a few feet wide; the dip needle shows high readings over only a very small area surrounding it.

The composition of the ore in the trench on lot 22, and the results obtained by Dulieux when he subjected a sample of it to magnetic concentration, are as

follows:-

It was crushed, then divided into three sizes by screening, and each size treated separately, yielding:—

	Crude ore			Concentrates			Tailings		
Size	Per cent of	Analysis		Per cent of	Analysis		Per cent of sized	Analysis	
	unsized	Fe	Ti	sized crude	Fe	Ti	crude	Fe	Ti
A: from 20 to 40 mesh	29.40	42.04	<b>5·4</b> 2	71.42	49.62	4.69	25.58	23 · 12	7.68
B: from 40 to 80 mesh	28.90	42.47	7.10	52.60	58 · 45	2.95	47.40	23 · 32	11.82
C: Through 80 mesh	$\begin{array}{c} 41.50 \\ 0.20 \end{array}$	41.21	4.29	53 · 42	53 · 40	2.33	46.58	29 · 43	5.16

Reference:—
Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1912, Que.
Bur. of Mines, 1913, pp. 78-84.
Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 95-100.

### Terrebonne County

### BERESFORD TOWNSHIP

Degrosbois Deposits.—Outcrops of titaniferous magnetite occur on lots 39, 40, and 41 in range VI, Beresford township, Terrebonne county. They lie within a few hundred feet of the railway track near Degrosbois station on the Montreal-Mont Laurier branch of the Canadian Pacific railway.

The Degrosbois titaniferous magnetite occurs in part of the same anorthosite mass in which the ilmenite deposits at the Ivry mine, two miles away, are found, but in a foliated gabbro phase of that rock that underlies the valley of the Rouge river near Degrosbois. Gabbro consisting of feldspar, pyroxene, and titaniferous magnetite, in which this last mineral is in places so plentiful as, perhaps, to constitute an ore, is exposed in a number of shallow pits and trenches on all three lots.

Judging by dip needle readings and the available exposures, the largest heavily mineralized area appears to be on lot 40, with a small extension over the boundary on to lot 41. It is about 500 feet long and 200 feet wide, and in the pits and trenches scattered over it are to be seen patches of mixed magnetite, pyroxene, and feldspar, the segregations ranging in size from those only a square foot or so in area to one whose dimensions are 47 by 27 feet.

A sample of the material from an outcrop on lot 40 yielded on analysis (Dulieux):—

A similar mineralized area, probably 70 feet long and having a maximum width of 27 feet, is found on lot 39. Samples of the ore taken from a trench 22 feet long and 5 feet deep, blasted out of one of the outcrops on this lot, yielded on analysis (Dulieux):—

	1	2
Iron Titanium	$40.76 \\ 4.49$	$44.04 \\ 5.09$

Another sample of ore, from a pile at the trench on lot 39, was pulverized until it would all pass through a 20-mesh screen, then separated into three sizes by screening, and each of these sizes was subjected to a process of magnetic separation in a small laboratory apparatus. The following table shows the composition of the crude ore and the effects of the magnetic separation:—

Analy	sis of Cru		Iron Titaniun	1		42.85 per cent 6.73 per cent			
		С	rude Ore		1	Concent	rates and	Tailings	
Size	Per cent	Fe	Ti	S	Р		Per cent	Fe	Ti
<b>A</b> : 20 to 40 mesh	9.16	49.50	4.37	0.68	0.064	Concentrate Tailing	89-11	51·32 27·30	3·69 13·57
B: 40 to 80 mesh	27.57	43.25	6.27	0.34	0.061	Concentrate	74.50	54·60 15·28	6·65 5·52
C: Through 80 mesh	63.27	41.85	7.25	1.09	0.067	Tailing		47.32	7.28

References:--

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1912, Que. Bur. of Mines, 1913, pp. 72-78.

Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 88-95.

# Beauce County

The occurrence of titaniferous magnetite in serpentine in the seigniory of St. Francois de Beauce is noted by T. Sterry Hunt, in the Geology of Canada, 1863. More recently, prospect workings along the eastern flank of the range of wooded hills that form the watershed between the river Des Plantes and the Caldwell river, two small tributaries that flow into the Chaudiere river from the northeast, have disclosed the mineral in two places—one in the unsurveyed Bloc range, the other in the St. Charles range. The two occurrences are less than a mile apart, on the west side of the valley of the Des Plantes river, and it is possible to drive from Beauceville, about six miles distant, on the Quebec Central railway, to within a mile of either.

Neither occurrence seems to have any special economic importance.

St. Charles Range.—The exposures on the St. Charles range are on lots 300 and 301, where some prospecting, mainly for asbestos, was done by the Quebec Mines and Metal Company, the owners of the mining rights.

On lot 301, the only showings are some small pockets of magnetite a few feet in diameter, enclosed in a highly ferruginous greenstone (serpentine). A sample from one of these pockets yielded on analysis (Dulieux):—

On lot 300, a lenticular body of magnetite 10 or 12 feet wide, showing no definite walls but passing by gradual transition into country rock on either side, is exposed in a small open-cut. There are no exposures of bed-rock around the excavation, but the deposit seems to lie near the border of a much

shattered and decomposed pyroxenite or peridotite. An analysis of the magnetite from this pit yielded:—

34.70 per cent Iron......... 12.36 

A second excavation on this lot is now full of water. Some of the excavated material lying alongside it carries perhaps as much as 50 per cent magnetite.

Bloc Range.—The workings on the Bloc range consist of a series of shallow trenches, spread over an area measuring about 200 feet in its greatest dimension, in a little glade in the midst of the woods. Small irregular pockets and stringers of magnetite in serpentine country rock are to be seen in a number of the trenches. The mineralized area in which they occur is about 110 feet long by 40 feet wide. At many places the magnetite passes by gradual transition into the surrounding serpentine.

In some of the pockets the magnetite is chromiferous (1) in others titaniferous (2), as is indicated in the following analysis (Dulieux):-

	1	2
$\begin{array}{c} \text{Fe.} \\ \text{Ti.} \\ \text{Cr}_2\text{O}_3. \\ \text{S.} \\ \text{P.} \end{array}$	43.06 $0.09$ $9.86$ $0.075$ $0.045$	47·73 9·78 Nil

References:-

Dulieux, P. E., Report on Mining Operations in the Province of Quebec for 1912, Que. Bur. of Mines, 1913, pp. 94-100.

Les Minerais de Fer de la Province de Québec, Que. Bur. of Mines, 1915, pp. 101-106.

Hunt, T. S., Geology of Canada, 1863, p. 501. Mackay, B. R., Beauceville, map area, Quebec: Geol. Sur. Can. Memoir 127, 1921, p. 85.

## Hull and Pontiac Counties

Titaniferous iron ores, hematites as well as magnetites, are known to occur at a number of points in the valleys of the Ottawa and Gatineau rivers, in Hull (formerly Ottawa) and Pontiac counties, Quebec. The occurrences are all small and unimportant.

#### TEMPLETON TOWNSHIP

Lot 28, Range VI.—Some of the hematite at the old Haycock mine, which supplied ore to bloomeries operated in Hull in 1873 and 1874, carries a certain amount of titanic acid (TiO2).

A sample from an ore pile remaining at one of the largest of the old workings, on the northeast part of lot 26, range VI, of Templeton township, yielded on analysis:-

65.56 per cent 3.52

On the top of a bluff close by there is a small lenticular body of hematite seven feet long and 20 inches wide at its centre—a sample from which gave:-

Fe	61.15 per cent
$\text{TiO}_2$	8.10 " "
5	0.008 " "
P	0.001 " "

A sample from another small exposure, measuring 12 inches by 24 inches, and lying 1,200 feet southwest of the foregoing, yielded:—

Fe	66.92	per cent
$\operatorname{TiO}_2$	5.95	66 64
S	0.036	66 69
P	0.01	66 66

#### HULL TOWNSHIP

Lot 1, Range XI.—On the south half of lot 1, range XI, Hull township, small stringers and lenses of titaniferous magnetite occur paralleling the lamination of a reddish gneissoid rock cut by pyroxene dikes.

The ore also is lamellar and, when broken across, shows on the fractured surface the edges of the thin interlaminated plates of ilmenite, as fine lustrous lines against the duller black of the magnetite. A sample of this material yielded on analysis:—

Fé	 58.21 per cent
TiO2	 16.80 " "

Lot 3, Range X.—On the south part of lot 3, range X, Hull township, a crooked, irregular vein of hematite, having an average width of about 10 inches, has been exposed for a length of 60 feet.

A sample of this vein yielded on analysis:-

Fe	 	50.98 per cent
TiO2	 	13.58 " "

#### BRISTOL TOWNSHIP

Lot 22, Range I.—An outcrop about two feet square, of a coarse-grained rusty hornblende rock carrying titaniferous magnetite in disseminated grains and patches, occurs on lot 22 in the first range of Bristol township. A sample from this outcrop yielded:—

Fe	 		 cent							
TiO						 	 	 	11.78	 

## CLARENDON TOWNSHIP

Lot 27, Range VII.—Titaniferous magnetite mixed with rock occurs in small veins and pockets in a coarse, dark hornblendic-looking rock on lot 27, range VII, of Clarendon township. A specimen yielded on analysis:—

Fe	 	 	 	7.23 " "
$TiO_2$	 	 	 	1.43

#### LITCHFIELD TOWNSHIP

Lot 12, Range V.—Many years ago a shaft 20 feet deep was sunk on what appears to have been a pocket of titaniferous magnetite associated with a coarse, dark, ferromagnesian-rich rock on lot 12 in the fifth range of Litchfield township. A sample of the coarsely crystalline ore yielded:—

E'e	·		 	 	 	 	 	 	 55.98	A.		
	$O_2$								13.03	**	**	

Lot 10, Range VIII.—Many loose scattered blocks of seemingly pure magnetite, and a single outcrop showing magnetite in place, occur on lot 10, range VIII of Litchfield township. A sample yielded on analysis:—

Fe	 	 	 	 	 	0 0	 	 	0 0	15.75 " "
$TiO_2$ .										

#### OTHER LOCALITIES

Other localities are on lots 4 and 5, range X, in Litchfield, and lots 12 and 13, range VI, in Sheen townships.

Reference:-

Cirkel, Fritz, Report on the Iron Ore Deposits along the Ottawa and Gatineau Rivers, Mines Branch, Pub. 23, Ottawa, 1909, pp. 61-69, 90-97.

## PROVINCE OF ONTARIO

## Leeds County

## TOWNSHIP OF NORTH CROSBY

Matthews or Yankee mine.—Some sixty-odd years ago, what is sometimes called the Matthews mine was opened on a deposit of magnetite on lot 1, concession VI of the township of North Crosby, Leeds county, Ontario, the scene of operation being the north shore of Mud lake on the Rideau canal, about a mile west of the village of Newboro' on the Brockville and Westport branch of the Canadian National railway.

Iron ore was mined here as early as 1860, and, in 1871, upwards of 4,000 tons were raised, of which 3,300 tons were shipped via the Rideau canal and Kingston to blast furnaces at Cleveland, Ohio. Further details concerning these mining operations, which have long since ceased, are not now available.

A single pit 300 feet long, about 100 feet wide, and which was reported to be 40 feet deep in 1871, constitutes the old workings. Filled with water it is now transformed into a horse pond in a pasture field. No ore in place is to be seen in its vicinity, all that now remains visible being fragments scattered through the remnants of the old mine dumps. A few small exposures of bed-rock that outcrop around the edges of the pit and in the surrounding field consist of rusty, friable, disintegrating gabbro carrying scattered grains of magnetite. The country rock is noticeably foliated, the strike of the laminations being about ENE. and parallel to the longer diameter of the pit. The dip is to the north, at a steep angle. The ore-body appears to have been a segregation lying with its greatest dimension conforming with the foliation of the enclosing rock.

Though no ore is now to be seen in the old working, a magnetic reconnaissance demonstrates the presence of an area of strongly magnetic attraction extending for some distance south and southwest of the pit. Another area of strong magnetic attraction, approximately 15,000 square feet in extent, exists about 600 feet northwest of the pit, and it is possible that a more extended survey would result in the discovery of other such areas on the surrounding farm land.

The ore remaining in the old dumps is a hard crystalline magnetite with sub-metallic lustre, mixed to some extent with black ferromagnesian minerals that require close inspection to distinguish them from the magnetite, and traversed by little veins or cracks, filled with a dead black, fine-grained substance that is probably magnetite also. Grains of pyrite occur scattered sparingly through the whole.

The chemical composition of the ore as indicated by the analyses of two samples taken from the dumps in 1919 and analysed in the Mines Branch laboratory by H. A. Leverin, is as follows:—

Fe	48·32 16·40
$\mathrm{SiO}_2$	6.84
P	0.048
0.66	0.159
V <sub>2</sub> U <sub>5</sub>	
C1203	
Ni Nil	* * * * * *
ZVAL	

An analysis published by Isidor Walz<sup>1</sup> in 1876 and cited by Kemp<sup>2</sup> shows 0.31 per cent V<sub>2</sub>O<sub>5</sub>.

## TOWNSHIP OF SOUTH CROSBY

Chaffey Mine.—The old Chaffey mine workings are at the east end of an island about 600 feet long and 400 feet wide that lies not far from the north shore of Mud lake. They are on lot 27 concession VI of the township of South Crosby and approximately half a mile south of the Matthews mine. The geological conditions at the two places are identical.

About 6,000 tons of iron ore were raised and shipped from the Chaffey mine in 1858 and 1859, 3,500 tons in 1871, and 14,520 tons from the Chaffey and Matthews mines combined during the years 1870 and 1871. The ore is said to have been sent to Pittsburg and Chicago to be used in puddling furnaces, and to Cleveland, Ohio.

As at the Matthews mine, the workings, long since abandoned, are now full of water. They consist of three large pits lying close together with their longer axes parallel. The ore appears to have been loaded direct from the pits into barges or scows moored at the east end of the island. The pits are separated from each other by walls of barren rock and are apparently on isolated ore-bodies. They average about 150 feet in length, are each about 50 feet wide at the surface, and are said to have attained a depth of about 50 feet. One or two prospect pits on other parts of the island do not seem to have proved any ore-bodies.

The ore-bodies occur in the same gabbro mass that contains the Matthews mine. This gabbro, which is for the most part granitoid in structure, becomes distinctly gneissoid near the ore, the general trend of the lamination being parallel to the longer dimensions of the pits. There is no sharp contact between ore and rock; the ore near the boundary of the deposits becomes leaner and leaner until it disappears in the normal gabbro. They seem to represent irregular, detached bodies or local concentrations of magnetite in a basic igneous rock.

Excepting a little in spots around the edges of the pits, no ore in place is visible at the workings, but observations with the dip needle would indicate that the deposits on the island have not been entirely exhausted.

<sup>1</sup> Amer. Chemist, June, 1876, p. 453.

<sup>&</sup>lt;sup>2</sup> U.S. Geol. Sur., 19th Ann. Rept., pt. 2, p. 392.

Analyses of ore from the Chaffey mine, the first of which (1) is given by Hunt, the second (2) by Pope, and the third (3) and fourth (4) of which were made in the Mines Branch laboratory by H. A. Leverin in 1919, are as follows:—

Annual State of Contract of Co	1 .	2	.3	4
Fe.	50.23	49.9	53.70	50·39 7·47
TiO <sub>2</sub>	9·80 7·10	10.21 $7.52$ $4.45$	11.36	6.38
Al <sub>2</sub> O <sub>3</sub>	5·65 4·5	2·17 3·13		
MgO		$\begin{array}{c} 0.52 \\ 0.22 \end{array}$		
P	0·08 1·52	0·03 0·82	$0.05 \\ 0.56 \\ 0.18$	0·05 1·08
V <sub>2</sub> O <sub>5</sub>		$0.35 \\ 0.11 \\ 0.23$	Trace	
MnO NiO		0.23	0.07	
Moisture	2.45	0.37		

References :-

Hunt, T. S., Geology of Canada, 1863, p. 674.
Hunt, T. S., Geol. Sur. Can., Report of Progress, 1866-69, pp. 284-285 (French ed.).
Vennor, H. G., Geol. Sur. Can., Report of Progress, 1871-72, pp. 121-122.
Ingall, E. D., Geol. Sur. Can., Annual Report, vol. XIII, 1900, pp. 66 I-69 I.
Pope, F. J., Trans. Amer. Inst. Min. Engrs., vol. 29, 1899, pp. 377 and 380.
Haanel, B. F., Mines Branch, Summary Report, 1909, p. 112.

# Frontenac County

## TOWNSHIPS OF HINCHINBROOKE AND BEDFORD

Eagle Lake, or Blessington Mine.—On lots 29 and 30 in the first concessions of both the townships of Hinchinbrooke and Bedford, titaniferous magnetite occurs in a number of pits from which apatite was once mined. The main pits are in Hinchinbrooke township, near the southwest shore of Eagle lake, and are separated from those in Bedford by the concession road between the two townships. The property, which as a whole is known as the Eagle Lake, or Blessington, mine, is a mile and a half from Tichborne station on the Kingston and Pembroke branch of the Canadian Pacific railway.

The magnetite, which is associated with apatite and much pyroxene, is found in and near the margin of a gabbro mass where the latter is in contact with the characteristic gneiss of the region.

### HINCHINBROOKE TOWNSHIP

Several openings have been made on a large apatite-bearing vein, or zone, near the southwest shore of Eagle lake, in Hinchinbrooke township, where the deposit is chiefly remarkable for the large development of magnetite. This mineral occurs both in small veins irregularly traversing the phosphatic zone, and also along with the apatite in the main lead. In one of the main pits the entire vein filling at one point is said to have consisted of magnetite, to the

complete exclusion of apatite, and it appears to have been not uncommon for one mineral suddenly to give place to the other. The veins of magnetite, though numerous, are much too small to be exploited.

About 700 tons of magnetite, taken out when mining apatite, were shipped

to the Hamilton Blast Furnace Company.

The magnetite is titaniferous, as will be seen in the following analysis made in the Mines Branch laboratory by H. A. Leverin on a sample collected from the old waste dumps in 1919:—

Fe	65.55
TiO <sub>2</sub>	4.97
P	0.273
S	0.110
$V_2O_5$	0.10
$\operatorname{Cr}_2\operatorname{O}_3$	0.05
Ni	0.015

#### BEDFORD TOWNSHIP

There are also a number of small openings across the road, in Bedford township, the deepest of which is not over 15 feet. Exposures of magnetite are to be seen in several of these, that mineral in small masses being most abundant in pyroxene or around its margin. Apatite in the form of grains, crystals, and masses is associated with much of the magnetite, but where the latter carries no visible apatite its phosphorus content is small.

A sample of the magnetite from Bedford township, which is beautifully crystalline and appears to be perfectly homogeneous when examined under the microscope, yielded on analysis (Pope):—

$Fe_2O_3$	62.39 \ Equivalent
FeO	26.93 \ to 64.58 Fe
TiO <sub>2</sub>	6.41
$SiO_2$	1.47
$Al_2O_3$	0.67
MgO	0.33
CaO	0.72
$P_2O_5$	0.03
S	0.06
$V_2O_5$	0.23
MnO	0.45
NiO	0.22
CoO	0.05

References:---

Miller, G. W., Ont. Bur. of Mines, Ann. Report, vol. 8, 1899, p. 167.

Pope, F. J., Trans. Amer. Inst. Min. Engrs., vol. 29, 1899, pp. 378 and 380.

Spence, H. S., Phosphate in Canada, Mines Branch, Pub. 396, 1920, pp. 44-45.

# Renfrew County

## TOWNSHIP OF BLITHFIELD

Lot 13, Concession I.—About three miles south of Calabogie station, in a side rock cut on the Kingston and Pembroke branch of the Canadian Pacific railway, a vein of magnetite dipping 35 degrees to the east shows for a length of about 75 feet. The height of the exposure is eight feet, but the foot-wall is not exposed, so this does not represent the full thickness of the deposit. The occurrence is on lot 13 in the first concession of Blithfield township, Renfrew county.

An average sample of the exposed portion of the deposit yielded on analysis:—

Fe	38.80 per cent
${ m TiO}_2$	4.96 " "
S	0.176 " "
P	0.013 " "
Insoluble	37.40 " "

Reference:-

Fréchette, H., Mines Branch, Summary Report, 1909, p. 86.

#### TOWNSHIP OF GRATTAN

Parks Property: Lot 16, Concession VIII.—Outcrops of an iron-bearing band, in places 50 feet wide, can be traced for 1,300 feet on lot 16, concession VIII, of the township of Grattan. The iron ore mineral, which is magnetite, is said to carry considerable titanium; the country rock is gneiss.

Reference:—
Gough, P. A., and Croze, W. W. J., Iron Ore Occurrences in Canada, vol. II, Mines
Branch, Pub. 217, 1917, p. 124.

### TOWNSHIP OF HORTON

Concession VI.—Just west of the Ottawa river, in the township of Horton, there is a considerable area of dark grey gabbro made up of feldspar and augite in about equal proportions and containing several small deposits of titaniferous magnetite. One of these, on the sixth concession, appears to be homogeneous throughout and shows a fairly well-defined contact with the enclosing rock.

A sample from this deposit yielded on analysis:—

$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$F_2O_3$	29.40 ] - 40.70
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		29.78 Fe 43.72
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$\mathrm{TiO}_2$	17.23
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		7.82
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		$3 \cdot 20$
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		5.67
$egin{array}{cccccccccccccccccccccccccccccccccccc$		$3 \cdot 42$
$egin{array}{cccccccccccccccccccccccccccccccccccc$		0.61
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		0.17
$egin{array}{cccccccccccccccccccccccccccccccccccc$	$v_2o_5$	0.63
$egin{array}{cccccccccccccccccccccccccccccccccccc$	MnO	0.22
$egin{array}{cccccccccccccccccccccccccccccccccccc$	NiO	0.43
$egin{array}{cccccccccccccccccccccccccccccccccccc$	CoO.,,	0.10
S 0.06	$P_2O_5\dots\dots\dots\dots\dots\dots\dots\dots$	0.14
Moisture	S	0.06
	Moisture	0.38

Reference:-

Pope, F. J., Trans. Amer. Inst. Min. Engrs., vol. 29, 1899, pp. 379 and 380.

# Hastings County

# TOWNSHIPS OF TUDOR AND LAKE

Orton Mine.—During the years 1912 and 1913, the Tivani Steel Company, of Belleville, Ont., mined a few tons of titaniferous magnetite on lots 56 and 57 west of the Hastings road, in Tudor township, Hastings county, Ont. The property, which is also known as the Orton mine, is four miles across country west of McDonald's siding on the Central Ontario branch of the Canadian National railway, and five or six miles by road from Millbridge village on the same railway.

The ore was shipped to Belleville to be used experimentally for the direct production of steel in the company's electric furnace—experiments that are stated to have been technically, if not commercially, successful.

The development work on the property consists of some surface strippings, three small open-cuts, and a shaft 32 feet deep. The ore was obtained from the open-cuts. The shaft is sunk in rock, but a drill hole ending 10 feet below the bottom of the shaft is said to have penetrated ore. A diamond drill-hole put down on the property about 1885 is said to have ended in ore at a depth of 70 feet.

The magnetite is found in small segregated bodies surrounded by particularly basic phases of the massive gabbro-diabase that constitutes bed-rock in the district. Judging by the outcrops and the results of a magnetometric survey of the property, made in 1915, these segregations of magnetite are confined to an area about 700 feet long and 400 feet wide, on the two lots. The largest individual segregation does not appear to be more than 150 feet long nor more than 40 or 50 feet in maximum width. From this size they grade down to insignificant patches a few square feet in extent. The uncertain and pockety nature of their occurrence is illustrated by the accompanying magnetometric map (in pocket).

All the magnetite is more or less mixed with black ferromagnesian minerals, even those portions that appear on casual inspection to be solid ore, and there is no sharp line of definition between the ore and the wall-rock.

The first of the following analyses of the magnetite from the Orton mine (1) was published by F. J. Pope; the second (2) by J. W. Evans; and the last two (3 and 4) were made in the Mines Branch laboratory, by H. A. Leverin:

	1	. 2	3	4
$Fe. \\ TiO_2. \\ SiO_2. \\ Al_2O_3. \\ MgO. \\ CaO. \\ Na_2O. \\ K_2O. \\ V_2O_5. \\ Cr_2O_3. \\ MnO. \\ NiO. \\ CoO. \\ P (P_2O_5). \\ S. \\$	48·54 8·17 10·37 5·01 4·23 4·40 0·57 0·12 0·29 0·12 0·31 0·26 0·04 0·09 0·04	0.2 0.39 0.041 Trace	54·72 13·89 0·34 Trace 0·11 0·081 0·237	49·90 11·83 6·66

Chapman, E. J., Trans. Roy. Soc. of Canada, sec. 3, vol. 3, 1885, p. 13.

Adams, F. D., and Barlow, A. E., Geology of the Haliburton and Bancroft areas, Geol Sur. Can., Memoir 6, 1910, p. 363.

Pope, F. J., Trans. Amer. Inst. Min. Engrs., vol. 29, 1899, pp. 379 and 380.

Evans, J. W., Jour. Can. Min. Inst., vol. 15, 1912, p. 128.

Robinson, A. H. A., Mines Branch, Summary Report, 1915, p. 37; also Mines Branch Map, 405, 1916.

Ricketts Property.—Pockety segregations of titaniferous magnetite, similar in every respect to those on the Orton mine, are also found on the immediately

adjoining lots 16 and 17 in the twelfth concession of the township of Lake. Though a magnetic survey (see map of Orton mine) indicates that the area over which these occur is somewhat more extensive than that on the Orton mine, some half-dozen or more exposures on lot 17, the Ricketts property, shows the ore to be somewhat leaner and the deposits more irregular.

A sample from one of the pits on lot 17 was analysed in the Mines Branch laboratory by H. A. Leverin, yielding:—

Fe	46.25 per cent
$TiO_2$	17.53 " "
$SiO_2$	2.10 " "
P.,.,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	0.016 " "
S	0.037 " "

References:-

Haanel, B. F., Mines Branch, Summary Report, 1909, p. 114.

Robinson, A. H. A., Mines Branch, Summary Report, 1915, p. 37; also, Mines Branch map 405, 1916.

Lots 41, 42, 54, and 55, Hastings Road, Tudor Township.—Southeast of the Orton and Ricketts properties, in the same gabbro mass, titaniferous magnetite has also been found on lots 41, 42, 54, and 55 east of the Hastings road and on lot 55 west of it. The country is bush-covered, exposures are few and small, and very little work of any kind has been done. So far as they are visible the occurrences are lean and, individually, small.

A special, picked sample from lot 54 (1) and a general sample from an exposure on lot 42 (2) were analysed in the Mines Branch laboratory by H. A. Leverin, with the following results:—

	1	2
FeTiO <sub>2</sub> SiO <sub>2</sub>	59·85 13·28	40·75 8·50 14·00
S P	Trace 0.035	0·287 0·028
$egin{array}{c} V_2O_5. & & & & \\ Cr_2O_3. & & & & \\ Ni. & & & & \end{array}$	Trace Trace	

Reference:-

Robinson, A. H. A., Mines Branch, Summary Report for 1915, pp. 37-38.

#### TOWNSHIP OF WOLLASTON

Lots 9 and 10, concession XV.—Titaniferous magnetite occurs disseminated through a large body of intrusive gabbro on lots 9 and 10 in the fifteenth concession of Wollaston township, Hastings county.

Reference:-

Lindeman, Einar, Magnetite Occurrences along the Central Ontario Railway, Mines Branch Pub. 184, p. 23.

## Haliburton County

## TOWNSHIP OF GLAMORGAN

Pine Lake, or Pusey, deposit.—The southeast corner of the township of Glamorgan, in Hastings county, Ont., is occupied by a great gabbro mass, characterized by a marked differentiation of the magma, giving rise to a great variation in the composition of the rock in different parts of the occurrence. The rock, which is on the whole rich in ferromagnesian constituents, varies from a

variety in which plagioclase predominates, through increasingly basic varieties, to a pyroxenite, or even to iron ore, as at the so-called Pusey, or Pine Lake mine on lot 35, concession IV. In this locality such an ultra-basic phase of the gabbro constitutes part of the ridge of low hills lying just south of Laronde creek, about three miles and a half from the village of Gooderham on the Irondale and Bancroft branch of the Canadian National railway. It is often cited as one of the largest bodies of titaniferous magnetite in Canada and pictured as an enormous deposit that—

....rises abruptly in the form of a succession of ledges to a height of from 80 to 100 feet above the general level of the ground, and is exposed in an easterly and westerly direction over a length of at least 1,800 feet, with an average width of 140 feet.

Such a description is decidedly misleading and appears to have arisen through confusing the dimensions of the pyroxenite mass in which it occurs with those of the deposit itself. Though a very considerable body of pyroxenite occurs at the Pusey mine, at only one place on the ridge, where a little rock was blasted out of the hillside many years ago, is magnetite exposed in sufficient quantity to be called ore even by courtesy. The other outcrops in the vicinity show only black pyroxene and hornblende, or gabbro carrying perhaps a little disseminated magnetite; and a hasty reconnaissance with the dip needle failed to show the presence of any appreciable magnetic attraction except in the immediate vicinity of the little pit.

Adams and Barlow found that in specimens which were supposed to represent the average character of the ore magnetite constituted only about one-fourth of the whole.

Analysis (1) of a specimen of the magnetite from the Pine Lake deposit, by Pope; and two analyses (2 and 3) of material from the pit, made in the Mines Branch laboratory by H. A. Leverin, are as follows:—

-	1	2	3
7e	44.1	31.01	41.91
0	13.52	7.54	14.91
$\Gamma$ i $O_2$ $S$ i $O_2$ $S$ i $O_3$ $S$ i $O_4$ $S$ i $O_5$ $S$ i $O_7$ $S$ i $O_8$ $S$ i	10.77	4.08	
$A_{19}O_{3}$	4.61		
CaO	4.84		
MgO	2.34		
7205	0.92		$0.04 \\ 0.03$
$\mathrm{Cr}_2\mathrm{O}_3.\ldots$	0.37		0.03
MnO	0.07		Nil
NiO	0.07		
CoO	0.31		
Na <sub>2</sub> O	0.24		
K <sub>2</sub> O	0.009	0.067	0.076
	.0.11	1.388	0.082

References:—
Pusey, C. J., Report of Royal Commission on Mineral Resources of Ont., Toronto,

Pusey, C. J., Report of Toyar Commanda, Sec. 3, vol. 3, 1885, p. 13.

Chapman, E. J., Trans., Roy. Soc. of Canada, Sec. 3, vol. 3, 1885, p. 13.

Pope, F. J., Trans. Amer. Inst. Min. Engrs., vol. 29, 1899, pp. 378 and 380.

Adams, F. D., and Barlow, A. E., Geology of the Haliburton and Bancroft Areas;

Geol. Sur. Can., Memoir No. 6, 1910, pp. 153-156 and 353.

Robinson, A. H. A., Mines Branch, Summary Report, 1919, p. 14.

<sup>1</sup> Trans. Roy. Soc. Can., Sec. 3, for 1884, p. 159, and for 1885, p. 13. Quoted by J. F. Kemp in "A brief review of the titaniferous magnetites," in School of Mines Quarterly, vol. 20, 1898-99, p. 329.

#### TOWNSHIP OF MINDEN

Lot 11, Concession I.—Rusty gneissic rock containing narrow stringers and disseminated grains of magnetite is exposed over an area about 4 feet wide and 40 or 50 feet long, on lot 11, concession I, of the township of Minden. The country rock of the district is crystalline limestone interstratified with well-banded gneiss and associated with coarse pegmatite.

A sample of the magnetite from these stringers, analysed in the Mines

Branch laboratory, yielded:-

Fe	42	.75
${ m TiO}_2\ldots\ldots\ldots$	0	.33
$SiO_2$	4.4	.46
P.,	0.	034
S		.103

Magnetite mixed with ferruginous silicates in gneiss and limestone from another exposure, on the lake shore on the same lot, is reported by Adams and Barlow to be free from titanium.

Chapman states that a sample of magnetite from this locality, analysed by him, yielded 25.51 per cent of TiO<sub>2</sub>.

References:-

Adams, F D., and Barlow, A. E., Geology of the Haliburton and Bancroft Areas; Geol. Sur. Can., Memoir No. 6, 1910, p. 359. Chapman, E. J., Trans. Roy. Soc. Can., sec. 3, vol. 3, 1885, p. 13.

# Parry Sound District

## TOWNSHIP OF LOUNT

Scattered over considerable areas in the township of Lount, in the Parry Sound district, are numerous small deposits of magnetite, some of which are titaniferous.

The country rock is gneiss, usually hornblendic, interbanded with crystalline limestone and traversed by narrow pegmatite dikes. The rock immediately associated with the magnetite is usually massive and coarse-grained and consists for the most part of hornblende, pyroxene, garnet, and epidote in variable proportions. There may be also a little white feldspar, calcite, mica, and pyrite mixed with it. Some of the reputed iron deposits consist almost entirely of masses of black, granular garnet.

Lot 132, Rousseau and Nipissing Road.—A small pit beside the Rousseau and Nipissing road on lot 132 contains some magnetite, iron-magnesia silicates, and much garnet, together with a little black mica, quartz, feldspar, and pyrite. The amount of magnetite exposed is inconsiderable; a sample of it analysed in the Mines Branch laboratory gave:—

Fe	53.47
TiO <sub>2</sub>	10.95
P	0.076
S	0.980
$V_2O_5$	0.23
$\mathrm{Cr}_2\mathrm{O}_3$	Nil
Ni	0.07
	0.01

Lot 144, Rousseau and Nipissing Road.—A sample of magnetite, containing also considerable garnet, some quartz, and a little pyrite, from a small exposure some distance west of the road, on lot 144, yielded:—

Fe	46.46
TiO <sub>2</sub>	
$\mathrm{TiO}_2$	11.50
$\operatorname{SiO}_2$	9.16
Q	
S	1.53
P	0.015

Lot 136.—A specimen from a small outcrop just east of the road, on lot 136, showed on analysis:—

Fe	71.36
${ m TiO}_2\ldots\ldots\ldots\ldots$	0.31
P., ., ., ., ., ., ., ., ., ., ., ., ., .	0.117
S	Trace
$ m V_{2}O_{5}\dots$	0.34
$\operatorname{Cr}_2\operatorname{O}_3$	Trace
Ni	0.03

Reference :-

Robinson, A. H. A., Mines Branch, Summary Report, 1916, pp. 17-18.

# Nipissing District

### OTTERTAIL BROOK

Some years ago, mining claims which had been staked on showings of magnetite found in the unsurveyed territory near the headwaters of Ottertail brook were surveyed for Mr. D. A. Dunlap, of Pembroke. When it was subsequently ascertained that the ore was highly titaniferous, the claims were abandoned.

The deposits are about five miles east of Bushnell station on the Temiskaming and Northern Ontario railway, on the old (unused) Timiskaming colonization road.

Reference:-

Stull, W. W., O.L.S., Sudbury, Ont., Personal communication.

#### MOUNTAIN LAKE

A group of mining claims was staked some years ago on outcrops of titaniferous magnetite at Mountain lake in the Timagami Forest Reserve. They lie six miles south of Latchford, a village on the Temiskaming and Northern Ontario railway, near the boundary between the districts of Nipissing and Timiskaming.

Reference:—
Bolton, L. L., Iron Ore Occurrences in Canada, vol. 2, Mines Branch Pub. 217, p. 106.

# Sudbury District

## TOWNSHIP OF McNAUGHT

McVittie Locations: W. D. 275 and W. D. 276.—The McVittie iron prospect is on mining locations W. D. 275 and W. D. 276, in the township of McNaught, about five miles northeast of Nemegos station on the main line of the Canadian Pacific railway.

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B'ed-rock in this vicinity is chiefly syenite, which is often porphyritic. There is also some granite; and, in immediate association with the titaniferous magnetite occurrences, a dark basic rock, a thin section of which showed the mineralogical composition to be: hornblende, 35 per cent; quartz, 35 per

cent; orthoclase, 20 per cent; magnetite, 10 per cent.

In this black hornblende rock, more lustrous titaniferous magnetite occurs in pockets, which vary in size from that of a plum to one showing a horizontal cross-section 94 square feet in area. The magnetite everywhere in the body of the deposits appears to be uniform in texture and composition, but at their borders merges gradually into the surrounding rock. The surface exposures, supplemented by a magnetic survey, indicate that the magnetite outcrops represent small isolated segregations, some of which are connected with each other by lean magnetite-bearing rock.

Analyses (1 and 2) of typical samples from two of the more prominent outcrops of magnetite, and (3) of the adjoining magnetite-bearing rock, yielded:—

	1	2	3
Iron Titanium dioxide (TiO <sub>2</sub> )	$\begin{array}{c} 63 \cdot 5 \\ 12 \cdot 5 \end{array}$	51·81 11·91	8·81 0·31

They show, as do analyses from many other deposits, that the proportion of titanium to iron is relatively less in the iron-rich portions of such segregations, a fact which would lead to the assumption that the iron commences to crystallize out of the molten magma before the titanium.

References:-

Haanel, B. F., Mines Branch, Summary Report, 1909, p. 110. Bolton, L. L., Iron Ore Occurrences in Canada, Mines Branch Pub. 217, 1917, pp. 89-90.

# Thunder Bay District

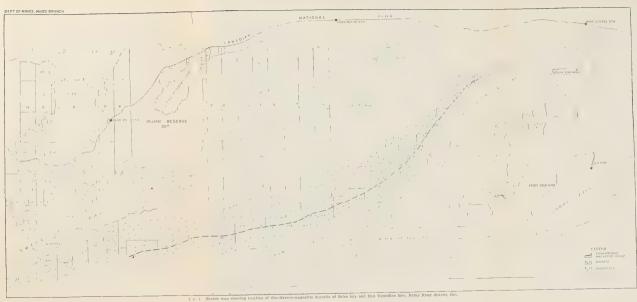
#### HAYSTACK MOUNTAIN

Some years ago, mining claims were staked around a high diabase hill, known as Haystack mountain, that rises like an island out of the swamp about three miles inland from the northeast corner of Windigo bay, lake Nipigon. The hill lies close to the Canadian National railway, about two miles west of Willet station. The claims were staked on account of local magnetic attraction that exists in the vicinity, which was thought to indicate the presence of a large hidden body of magnetite.

An examination of the diabase forming the hill shows it to contain numerous small patches of titaniferous magnetite, occurring as segregations in coarser portions of the diabase. The segregations are not confined to any particular part of the hill, but are scattered all through it, and are sufficient to account for the local magnetic attraction. There is no indication of the presence of magnetite deposits of any importance.

Reference:-

Moore, E. S., Ont. Bur. of Mines, 18th Annual Report, 1909, p, 162.





# Rainy River District

## SEINE BAY AND BAD VERMILION LAKE

The most extensive deposit, or rather series of deposits, of titaniferous magnetite yet known in Canada are those that extend, like a broken chain, in a narrow belt of basic gabbro, for 14 miles along the north shores of Seine

bay and Bad Vermilion lake in Rainy River district, Ontario.

The eastern end of the line of outcrops, which is about half way along the northwest shore of Bad Vermilion lake, can be conveniently reached from Mine Centre, a station on the Canadian National railway between Port Arthur and Winnipeg. A wagon road about a mile in length leads from Mine Centre to Bad Vermilion lake, where canoe or launch can be taken to outcrops along the shore, while a trail starting from the bottom of the southwest arm of the lake and leading to the east end of Seine bay runs nearly parallel with, and only a short distance from, the part of the titaniferous iron range lying between these two bodies of water. The western extremity of the range, where it outcrops on the north shore at the mouth of Seine bay, is accessible by gasoline launch from Fort Frances, twenty miles distant across Rainy lake; while farther east, where the outcrops are as much as half a mile inland, they may be reached at a number of points by old lumber roads running inland from the shore. The line of outcrops is nowhere more than six miles, and at the eastern extremity is only two miles from the railway. (See sketch map Fig. 4).

The rocks in the vicinity of the iron-titanium deposits comprise Keewatin greenstones, schists, slates, felsites, and agglomerates; and interstratified with the Keewatin series, though of later age, bands of coarse-textured gabbro of two types. One type, which is highly feldspathic, consisting almost wholly of basic feldspar, might be classed as an anorthosite; the other type is highly hornblendic, and, in places, might be called amphibolite. Certain marginal facies of the anorthosite on Bad Vermilion lake grade into dark gabbro, and this suggests that both types are differentiation products of a common magma. In some of their occurrences both types are thoroughly schistified, but for the most part they are massive and unsheared. Keewatin and gabbro are both

intruded by granite.

The titaniferous magnetite occurs as local ultra-basic segregations in a narrow belt of the dark hornblende-gabbro, the segregations varying both in the frequency of their occurrence in different parts of the zone, and in their size. Outcrops from a few feet to 100 feet and more in length, some of them separated by long drift-covered intervals, occur over the whole 14 miles, and exploration with the dip needle indicates the existence of numerous additional segregations hidden under the drift-covered intervals between. A typical segregation consists of a fairly well-marked central core of apparently homogeneous magnetite surrounded by a zone of leaner orey material that merges gradually into gabbro or amphibolite. In some, however, a distinct central core is lacking, and in others the zone of transition from ore to rock is quite narrow. The ore deposits are apparently flattened lenticular bodies, standing with their longer axes in a nearly vertical plane parallel with the schistosity of the rocks of the district. The gabbro of which they form part was probably injected into the Keewatin series before its chief deformation and metamorphism and thus partakes of its structural features.

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Though the occurrence of titaniferous magnetite in the vicinity of Seine bay has been known for a long time, and the deposits have been staked, abandoned, and re-staked several times, the only work done on them is a little diamond drilling, some trenching, and a magnetometric survey of a drift-covered portion of the range lying between Seine bay and Bad Vermilion lake.

Mining Locations 181 P, 182 P, and 183 P.—In 1911, some trenching and a little diamond drilling was done on outcrops that occur about half a mile inland from the north shore of Seine bay on mining locations 181 P, 182 P, and 183 P (see map Fig. 4); and the greatest continuous width of ore yet found on the range is said to have been uncovered in the trenches on 182 P. A band or lens at this point is said to have had a maximum thickness of 62 feet.

Numerous exposures of ore can still be seen in the old cross trenches and in natural outcrops scattered across the three locations, and widths of 20 and 30 feet of ore, with only one wall of the deposit exposed, can still be measured in some of the trenches; but earth washed in from the sides prevents the determination of the full width in most of them. Analysis No. 12 in the table of analyses given below is that of an average sample taken across 20 feet of outcrop on 182 P.

No information is available concerning the extent or results of the diamond drilling, which is said to have been done for a Mr. Hunter, of Duluth, Minn., in 1911.

Mining Locations A.L. 25, A.L. 26, A.L. 27, A.L. 28, and A.L. 29.—During the summer of 1917, the Mines Branch made a magnetometric survey of the five mining locations numbered consecutively from east to west A.L. 25 to A.L. 29, which lie between Seine bay and Bad Vermilion lake (see magnetometric map in pocket). At the same time some diamond drilling was being carried on by the owners of this portion of the range, under the direction of Dr. W. L. Goodwin, of Kingston.

The magnetometric survey indicates the existence of a nearly continuous succession of magnetic deposits extending in the general direction S. 60° W. from the east boundary of A.L. 25 to the west boundary of A.L. 27, a total distance of about 6,000 feet. For a further 6,000 feet, in the same general direction, across locations A.L. 28 and A.L. 29, the magnetic bodies are less numerous, with wider intervals between them.

The thickness of the individual segregations measured in various exposures ranges from 2 to 45 feet, there being included in the maximum measurement 20 feet of apparently solid magnetite and 25 feet of magnetite mixed with silicate minerals. The full width of the segregations, however, is exposed at only a few places.

Three diamond drill holes, one each on A.L. 26, A.L. 28, and A.L. 29, were put down on this portion of the range, by Dr. Goodwin. The hole on A.L. 26 was pointed to pass beneath an outcrop of solid magnetite 32 feet wide and, at a depth of 460 feet vertically below it, cut through 16 feet (horizontal width) of ore. Analyses of the ore both at the outcrop and in the drill hole are given in the table (Nos. 4, 5, and 18).

The location of the drill holes on A.L. 28 and A.L. 29 was not determined by the position of surface outcrops, but by that of areas of strong magnetic attraction that had been found with the magnetometer, on a sand plain. The holes were directed towards two such areas, and both encountered bodies of magnetite at the points indicated by the magnetic survey. The greatest thickness of ore cut in either hole was 17 feet of disseminated magnetite, in the hole on A.L. 29. Analyses and widths of other ore bands cut are given in the table of analyses (Nos. 19, 20, and 21).

A.L. 25 to A.L. 19.—The magnetic survey was not extended to cover the range east of A.L. 25, but numerous closely spaced outcrops demonstrate the continuity of the zone of iron-titanium-bearing deposits at least to A.L. 19, nearly a mile and a half farther east.

Diamond drill holes, one on A.L. 24 and one on A.L. 17, penetrated respectively 11 feet and 8 feet of magnetite, at vertical depths of 230 and 150 feet from the surface. A third, a shallow vertical hole on A.L. 19, was so placed that it yielded no conclusive results.

Associated with magnetite in the drill cores, usually as the filling in minute cracks and fissures, small amounts of the following minerals occur: quartz, calcite, siderite, pyrite, pyrrhotite, chalcopyrite, and epidote.

The following table gives the composition of a number of samples taken from surface exposures and diamond drill cores:-

Analyses of Titaniferous Magnetite from the Seine Bay Iron Range OUTCROP SAMPLES

Analysis No.	Fe	SiO <sub>2</sub>	TiO <sub>2</sub>	P	S		Analyst	
1	48 · 45 44 · 04 46 · 70 46 · 60 44 · 81 47 · 62 32 · 35 42 · 82 34 · 51 32 · 10 44 · 92 50 · 03 49 · 36	$7 \cdot 35 \\ 6 \cdot 76 \\ 6 \cdot 50 \\ 2 \cdot 47$ $3 \cdot 14 \\ 3 \cdot 00 \\ 12 \cdot 41 \\ 6 \cdot 66 \\ 12 \cdot 20 \\ 16 \cdot 00 \\ 5 \cdot 30 \\ 2 \cdot 40 \\ 3 \cdot 74$	$\begin{array}{c} 26 \cdot 03 \\ 17 \cdot 21 \\ 20 \cdot 50 \\ 27 \cdot 54 \\ \\ 20 \cdot 70 \\ 19 \cdot 29 \\ 9 \cdot 65 \\ 19 \cdot 28 \\ 14 \cdot 28 \\ 10 \cdot 71 \\ 17 \cdot 86 \\ 10 \cdot 34 \\ 15 \cdot 71 \\ \end{array}$	$\begin{array}{c} 0.05 \\ 0.004 \\ 0.02 \\ 0.029 \\ \hline 0.003 \\ 0.004 \\ 1.320 \\ 0.005 \\ 1.380 \\ 1.360 \\ 0.006 \\ 0.005 \\ 0.005 \\ \hline \end{array}$	$0.121 \\ 0.04 \\ 0.041$	H. A. Leverin, Analysis furnisl LeDoux & Co W. L. Goodw H. A. Leverin,	Mines Branched by Dr. V ., N.Y., fur	W. L. Goodwin.

1. Average of a ton sample from A.L. 17.

Average of a ton sample from A.L. 17.
 Average sample across 20 feet of outcrop on A.L. 17.
 Average of a bulk sample from A.L. 17 amd A.L. 18.
 and 5. Typical samples from 32 feet of outcrop, or the boundary between A.L. 26 and A.L. 27.

6. Average sample from 10 feet of outcrop on A.L. 25.

7. Average sample from a low grade outcrop on a.1. 26.
8. Average sample from 20 feet of outcrop on A.1. 26.
9. Average sample from 25 feet of outcrop on A.L. 26, lying alongside exposure from which

9. Average sample from 25 feet of outcrop on A.L. 25, lying alongside exposure from which previous sample (8) was taken.

10. Average sample from 12 feet of outcrop on A.L. 27.

11. Average sample from 12 feet of outcrop on A.L. 26.
12. Average sample from 20 feet of outcrop on 182 P (at west end of range).
13. Typical sample from an exposure between 100 and 150 feet long, and about 3 feet in average width, on H.P. 96 at the eastern extremity of the range.

# Analyses of Titaniferous Magnetite from the Seine Bay Iron Range —Concluded

#### DRILL CORE SAMPLES

Analysis No.	Fe	SiO <sub>2</sub>	TiO <sub>2</sub>	P	S	Analyst		
14	45.60	3.36	24.00	0.005		H. A. Leverin,	Mines Branch,	Laboratory
5	$24 \cdot 97$	29.82	$6 \cdot 75$	1.296	0.140		. 66	**
16	46.75	4.14	23 · 40	0.004	0.101	66	66	66
[7	44.64	3.40	18.00	0.004	0.635	66	66	66
18	45.50	4.50	24.00	0.004	0.371	46	"	"
19	35.38	8.24	18.60	0.008	0.405		66	46
20	30.85	12.26	11:40	2 · 405	0.100	66	66	66
21	39.43	3.68	$22 \cdot 20$	0.005	0.407	66	66	44
				İ				

- 14. Sample from drill hole on A.L. 17, across 8 ft. of ore at depth of 150 feet.
  15. Sample from drill hole on A.L. 17, at depth of 300 feet.
  16. Sample from drill hole on A.L. 26, across 14 ft. of ore at depth of 215 feet.
  17. Sample from drill hole on A.L. 26, across 12 ft. of ore at depth of 376 feet.
- 18. Sample from drill hole on A.L. 28, across 16 ft. of ore at depth of 460 feet.

  19. Sample from drill hole on A.L. 28, across 3 ft. of ore at depth of 365 feet.

  20. Sample from drill hole on A.L. 28, across 7.5 ft. of ore at depth of 375 feet.

  21. Sample from drill hole on A.L. 29, across 4 ft. of ore at depth of 230 feet.

It may be noted that the samples richest in iron—those carrying between 40 and 50 per cent—are almost without exception relatively lower in phosphorus than those in which the iron content is reduced below 40 per cent by intermixed rock matter, which would tend to show that the phosphorus is associated with the silicate minerals rather than with the iron-titanium segregate. Probably owing to leaching by surface water, the outcrop samples are on the average lower in sulphur than those from depth.

In addition to the above, four specially selected samples were analysed in the Mines Branch laboratory to ascertain the presence in them or otherwise of some of the rarer metals, with the following results:—

	Fe	TiO <sub>2</sub>	Р	S	V <sub>2</sub> O <sub>5</sub>	$Cr_2O_3$	Ni
<ol> <li>Diamond drill core from A.L. 26.</li> <li>Outcrop sample from A.L. 26.</li> <li>Outcrop sample from A.L. 27.</li> <li>Outcrop sample from H.P. 96.</li> </ol>	48.00 $52.21$ $37.51$ $57.67$	19·87 27·38 11·86 20·28	0.054 $0.032$ $2.608$ $0.058$	0·106 0·014 trace 0·007	trace nil trace 0·20	0.07 ril trace nil	nil nil nil trace

A shipment of 275 pounds of ore averaging 46.44 per cent iron and 28.07 per cent titanic acid (TiO2), from some of the outcrops on the shore of Bad Vermilion lake, was sent to the Mines Branch ore testing laboratory for treatment in 1918. The results, which are given below, show that it is not possible to effect a separation of the iron and titanium by mechanical means.

# Experiments on the Concentration of Titaniferous Magnetite from Bad Vermilion Lake

Test No. I.—Separation by means of a horseshoe magnet, with sheath on, under water.

Mesh	Wt. of ore taken	Product	Weight	Anal	ysis'
21,2 0012	grams	Troduct	products	Per cent   Fe	Per cent TiO <sub>2</sub>
20	2,000	Magnetic	790	50.05	24.40
38 .	. 1,000	Non-magnetic	186 154 47	32·29 50·45	40·15 24·20
80	1,000	Non-magnetic	161 38	31.86 $49.60$ $33.05$	39·75 25·20 38·35
150	1,000	Magnetic. Non-magnetic.	159 41	$   \begin{array}{r}     35.03 \\     49.38 \\     25.22   \end{array} $	32·08 35·80

Test No. II.—Separation by water concentration on sand table after crushing to 30 mesh.

70 1 4	Weight	Analysis		
Product	weight	Per cent Fe	Per cent TiO <sub>2</sub>	
Concentrate	1b. 82·0 16·0 4·0	46·30 42·26 35·79	29·67 24·20 24·00	

Test No. III.—Magnetic separation (wet) on the Ullrich magnetic separator, after crushing to 35 mesh.

	Wainb +	Ana	lysis
Product	Weight	Per cent Fe	Per cent TiO <sub>2</sub>
Ring No. 1	1b. 27·5 5·5 2·0 6·5 0·5 4·0	50.95 50.35 48.22 29.72 31.44 30.43	22.93 23.44 27.65 44.24 35.36 34.75

Lawson, A. C., The Archean geology of Rainy lake re-studied: Geol. Sur. Can., Memoir 40, 1913, pp. 35 and 42.

Robinson, A. H. A., The titaniferous magnetite deposits of Seine bay and Bad Vermilion lake: Mines Branch, Summary Report, 1917, pp. 11-22.

Parsons, A. L., Ont. Bur. Mines, 27th Annual Report, 1918, pp. 176-183.

Timm, W. B., Mines Branch, Summary Report, 1918, p. 144.

### PROVINCE OF ALBERTA

#### BURMIS

Deposits of titaniferous magnetite differing both in origin and mode of occurrence from those already described are found in the province of Alberta, near Burmis, a station on the Crowsnest Pass division of the Canadian Pacific railway, nine miles east of the coal mining town of Blairmore.

They consist of beds of indurated black magnetic sand, probably representing an ancient shore concentration, intercalated in a series of soft coarsegrained sandstones that outcrop along the foothills two or three miles east of the Livingstone mountain range. They have been inadequately prospected at places for a distance of eight miles north from Burmis, the greatest amount of work having been done on the more northerly exposures, on the Gardiner claims on the headwaters of Cow creek, where the existence of at least three beds of magnetite is indicated. Two of these beds show thicknesses of eight and one-half and ten and one-half feet respectively in the single opening on each in which they could be measured. Two incompletely exposed beds in some shallow surface workings about a mile north of Burmis have probable thicknesses of five and three feet respectively at that place. Beds of consolidated titaniferous-magnetite sands, occurring under similar geological conditions to those at Burmis, are also reported 80 miles to the southwest, in the state of Montana.1

Under the microscope the ferruginous beds are seen to consist of more or less rounded particles of magnetite, quartz, and augite, with a little secondary calcite, the whole cemented together by iron oxide.

Analyses of typical samples are as follows:-

# Analyses of Titaniferous Magnetite from Burmis, Alberta

	Fe	TiO <sub>2</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	V <sub>2</sub> O <sub>5</sub>	Cr <sub>2</sub> O <sub>3</sub>	NiO	Р	S
2 3	59·08 55·50 39·80 58·82	$5 \cdot 74 \\ 5 \cdot 56$	$12.53 \\ 18.33$	2•30	$2 \cdot 78$ $2 \cdot 21$	$egin{array}{c} 0\!\cdot\!52 \ 2\!\cdot\!25 \end{array}$		trace		$0.10 \\ 0.073$	

- 1. Sample from exposure one mile north of Burmis, collected by J. A. Allen, analysed
- by J. A. Kelso. 2. Sample from the 3-feet bed one mile north of Burmis, collected by W. W. Leach, analysed in Mines Branch laboratory.

  3. Sample from 10½-feet bed, eight miles north of Burmis, collected by W. W. Leach.
- analysed in Mines Branch laboratory.

  4. Specimen collected by W. W. Leach, in 1911, analysed in Mines Branch laboratory, by H. A. Leverin in 1921.

The titaniferous magnetite from Burmis carries too little titanium to be of interest for that element, but should further prospecting prove the ferrugin-

<sup>&</sup>lt;sup>1</sup> Stebinger, Eugene, Titaniferous magnetite beds on the Blackfeet Indian Reservation, Montana: U.S. Geol. Sur. Bull. 540-H, 1913, pp. 3-11.

ous beds to be of sufficient extent and thickness it may at some future time prove of value as an iron ore. The maximum amount of metallic titanium shown in the above analyses—407 per cent—should not be an insuperable obstacle to its use for that purpose should circumstances ultimately prove favourable otherwise.

### References:-

Leach, W. W., Geology of the Blairmore map area, Alberta: Geol. Sur. Can., Summary Report, 1911, pp. 199-200.

Allan, J. A., First Annual Report on the Mineral Resources of Alberta, Edmonton, 1920, pp. 62-65.



# PART III SOURCES, PRODUCTION, AND USES OF TITANIUM



# SOURCES, PRODUCTION, AND USES OF TITANIUM THE WORLD'S COMMERCIAL SOURCES AND PRODUCTION OF TITANIUM MINERALS

Until 1903, Norway was the only country producing titanium minerals in quantity, and the recorded production, all of it rutile, was at the rate of less than 100 metric tons annually. With the exploitation of the Virginia deposits, however, the United States, which previous to 1903 had produced annually only a few hundred pounds of rutile gathered in the fields of Chester county, Pennsylvania, for use in the artificial tooth industry and for cabinet specimens, became an important producer also; and by 1909 the annual production of the Virginia deposits had surpassed that of Norway and continued to do so until, in 1918, a sudden large increase in the Norwegian production put that country again in the lead. There has also been a few scattered shipments of rutile from Australia, and a few hundred tons of rutilebearing ilmenite has been exported from Canada (Quebec) for experimental purposes.

The first shipments of ilmenite officially recorded as such, in Canada, were made from St. Urbain, in the province of Quebec, in 1908. In 1913 the United States also became a steady producer of that mineral. No information

is available concerning the ilmenite production of Norway.

The magnitude of the world's production of titanium minerals—which is also a measure of the world's present demands-and the relative proportions heretofore furnished by the three producing countries, Norway, the United States, and Canada, are set forth in the following table:-

# Annual Production of Titanium Minerals in Norway, the United States, and Canada, from 1906 to 1920

**	Norway <sup>1</sup>	United	States <sup>2</sup>	Canada <sup>3</sup>	Wo	orld
Year	Rutile	Rutile	Ilmenite	Ilmenite	Rutile	Ilmenite
1906. 1907. 1908. 1909. 1910. 1911. 1912. 1913. 1914. 1915. 1916. 1917. 1918. 1919. 1919.	116 76 100 77 30 170 103 129 2,412	Short tons 41 118 16 121 566 ril 100 305 94 250 110 206 261 102 277	250 89 300 95 1,339 1,644 106 268	Short tons  250 820 3,233 3,616 1,185 5,360 nil nil 3,209 4,978 6,330 321 960	Short tons 102 179 107 121 694 84 210 390 127 437 224 348 2,920	Short tons  277 820 3,233 3,616 1,185 5,610 89 300 3,304 6,317 7,974
1940					*	na fan 1018

<sup>1</sup>The figures for Norway are taken from The Mineral Industry. Norwegian returns for 1918,

<sup>&</sup>lt;sup>2</sup>The United States figures for 1906, 1907, and 1903 are taken from Watson and Taber, Bull. III-A, Virginia Geol. Sur., p. 51; the remainder from the annual volumes of Mineral Resources of the U.S., published by the U.S. Geol. Survey.

The figures for Canada have been compiled from the Annual Reports of the Quebec Bureau of Mines and of the Division of Mineral Resources and Statistics, Mines Branch, Ottawa. 1919, and 1920 probably include ilmenite.

<sup>1</sup> Hess, F. L., Min. Res. of the U.S., 1908, p. 744.

The Norwegian rutile comes from Kragerö, in southern Norway, and is marketed in the form of black concentrates carrying 93 to 98 per cent titanic acid (TiO<sub>2</sub>). The deposits from which it is obtained are the largest known in Europe and probably the second largest in the world, the Virginia deposits being said to be more extensive. They are reported to be capable of maintaining for many years an output much in excess of the quantity that it has hitherto been found possible to dispose of.

The United States rutile, also, is marketed in the form of concentrates, lighter in colour than the Norwegian product and carrying 94 to 96 per cent TiO<sub>2</sub>. It is all produced by the American Rutile Company at their plant at Roseland, Virginia, from rock carrying 4 or 5 per cent rutile and about the same quantity of ilmenite. In Nelson and Amherst counties, Virginia, there is said to be a practically unlimited supply of very pure rutile that can be

concentrated to yield a high grade product2

In the present undeveloped condition of the Canadian rutile deposits at St. Urbain, Quebec, nothing very definite can be said about their commercial possibilities. The rutile itself is of excellent quality and is apparently in very considerable amount, but it is so irregularly distributed through masses of ilmenite as to render uncertain any estimate of the possibilities of the area as a dependable source for large supplies. A trial shipment of about 1,000 tons of the St. Urbain ore, sent to the General Electric Co's Lynn works in 1910, is said to have carried 6 per cent rutile.<sup>3</sup>

There have also been a few small sporadic shipments of rutile from Australia (South Australia and Queensland). A little very pure rutile from Queensland was sold in the United States in 1908 (Min. Res. U.S., 1908, p. 744). There is, however, no information available on which it is possible to base an opinion as to the possibilities of the Australian deposits as steady producers of rutile.

Special interest in ilmenite as a source of titanium compounds may be said to have arisen with the development of the electric furnace process for the production of ferrotitanium alloys in the opening decade of the present century. The first shipments from St. Urbain, Quebec, in 1908 and 1909, were of material from old dumps, which was sent to Niagara Falls, N.Y., to be tested for this purpose. Actual mining operations followed later, both at St. Urbain and at Ivry, in Terrebonne county; and the province of Quebec has supplied the bulk of the ilmenite heretofore used in the manufacture of ferrotitanium in the United States. Latterly, however, Quebec ilmenite has been to a large extent supplanted in the United States—at present its only market—by ilmenite concentrates from Florida and Virginia. The Canadian mineral which is marketed as mined—without any preliminary treatment—is not as high grade a material as the United States product.

Until 1916, all the ilmenite produced in the United States was obtained as a by-product of the concentration of rutile-bearing syenite at the American Rutile Co.'s plant at Roseland, Virginia. At the present time ilmenite is also being produced in Florida, by Buckman and Pritchard (Inc.), a concern under the same financial control as the Titanium Alloy Manufacturing Co. and the Titanium Pigment Co., of Niagara Falls, N.Y. The Buckman and Pritchard

<sup>2</sup> Bulletin III-A, Virginia Geological Survey, 1913, p. 266. <sup>3</sup> Hess, F. L., Min. Res. of the U.S. for 1910, p. 762.

<sup>&</sup>lt;sup>1</sup> Hess, F. L., Titanium: Annual volumes of Min. Res. of the U.S.

plant at Mineral City, four miles south of Pablo Beach, Fla., produces ilmenite. rutile, zircon, and monazite by washing the beach sands found there, their chief

product as regards quantity being ilmenite.

While no figures are available concerning ilmenite production in Norway, it seems altogether likely that a certain amount is obtained there as a byproduct of the concentration of rutile, and recently ilmenite has been mined for itself, for use in the manufacture of white pigments. It was stated in 19201 that the Titan Co.'s plant at Fredrickstad was using ilmenite for this purpose at the rate of about 4,000 tons annually.

In the light of our present knowledge, the world's most important ilmenite reserves are probably to be found in the province of Quebec, Canada, and in the Ekersund-Soggendal district in Norway. Of three deposits in the vicinity of Soggendal, south of Ekersund, on the southwest coast of Norway, one, Laxdalsfelterne, is estimated to contain 250,000 tons of ilmenite carrying about 35 per cent TiO<sub>2</sub>; a second, Blaafjeld, 100,000 tons carrying 40 to 45 per cent TiO<sub>2</sub>; and the third, Storgang, 3,000,000 tons carrying 25 to 40 per cent TiO<sub>2</sub>. It is from the last two that the Titan Co. secures its raw material.

The Quebec ilmenite deposits, at St. Urbain and at Ivry, compare favourably

both as to size and titanium content with those found in Norway.

As already stated, all the ilmenite produced in the United States is in the form of concentrates which carry 48 to 55 per cent TiO2, and is consequently a higher grade material than either the Norwegian or Canadian raw ores. At the American Rutile Company's mill, rutile-ilmenite-bearing syenite is crushed by stamps, classified in Brown classifiers, and thence delivered to Wilfley tables, which separate the ore into a first concentrate, middlings, and tails. The middlings are returned to the stamp battery for further crushing and the tailings go to waste, except when, as is sometimes the case, they consist of practically all feldspar, when they are sold to manufacturers of porcelain and pottery. The concentrates, which consist of rutile and ilmenite with small amounts of silicate minerals, are passed through a Wetherill magnetic separator to effect the removal of ilmenite. Rutile for use in the ceramic industries is further pulverized in Sturtevant emery rolls.1

At the Florida plant of Buckman and Pritchard, Inc., the raw material, a beach sand, is excavated by dredging and passed over concentrating tables without any preliminary treatment. The table concentrates consisting of ilmenite, rutile, zircon, and monazite are dried in a Ruggles dryer and then put through a Dings magnetic separator, which separates the ilmenite; the tailings from the separator are again passed over tables to separate monazite and zircon. The commercial products are zircon, monazite, and an ilmenite concentrate carrying about 52

per cent TiO2. A marketable rutile product could also be made.2

At Mineral City, Florida, there is said to be available 1,025,000 tons of beach sand, carrying, ilmenite 5.43 per cent; rutile 0.59 per cent; monazite, 0.08 per cent; and zircon, 1.96 per cent: together with a further 31,750,000 tons of dune sand, carrying, ilmenite 1.06 per cent; rutile 0.12 per cent; monazite 0.015 per cent; and zircon 0.46 per cent.

A notable deposit of ilmenite associated with uranium minerals o curs near Olary, in South Australia and has been worked in recent years as a source

<sup>1</sup> Chem. Trade Jour. and Chem. Engr., Sept. 25, 1920, p. 407.
2 Bull. III-A, Virginia Geol. Survey, 1913, pp. 169-172.
3 Liddell, D. M., A rare Florida mineral: Eng. and Min. Jour., vol. 104, 1917, pp. 153-155.

of radium compounds. A specimen of the ilmenite from this deposit yielded

51.85 per cent TiO<sub>2.1</sub>

Ilmenite also is the chief constituent of the crude monazite sands of Ceylon and Travancore, India, and these might be expected to yield ilmenite as a cheap by-product.2

A deposit of ilmenite estimated to contain 3,500,000 metric tons of ore is also said to have been discovered in Madagascar in 1912. Some samples from the

deposit indicated a tenor of about 40 per cent titanium dioxide.3

The use of ilmenite is confined almost entirely to the ferro-titanium alloy. the arc lamp electrode, and the titanium pigment industries. Titanium compounds for other purposes are derived preferably, if not necessarily, from rutile. For some unexplained reason the ilmenite produced by the American Rutile Company seems to be specially adapted to the manufacture of electrodes, and the Metal and Thermit Corporation has also found it best adapted to its process for making carbon-free ferrotitanium, consequently the Virginia product has commanded a special price—in 1918, \$25 per ton as compared with \$6.25 per ton for Florida concentrates. The Titanium Alloy Manufacturing Company, on the other hand, has found ordinary ilmenite suitable for its needs; through Buckman and Pritchard, Inc., it controls its own source of supply.<sup>4</sup>

Practically all the ilmenite produced in Quebec has gone to the Niagara Falls plant of the Titanium Alloy Manufacturing Company to be used in the manufacture of ferrocarbontitanium in the electric furnace. The value of the Canadian mineral at the mine, as deduced from the figures for output and aggregate values given in the annual reports of the Quebec Bureau of Mines,

averaged \$1.97 per ton in 1913 and \$3.12 in 1920.

The prices of titanium minerals, as quoted by the Foote Mineral Company in the Engineering and Mining Journal of January 7, 1922, are as follows: ilmenite, carrying 52 per cent TiO2, 1½ to 2 cents per lb. for ore; rutile, carrying 95 per cent TiO2, 12 cents per lb. for ore, with concessions on large con-On the same date ferrocarbontitanium, containing 15 to 18 per cent titanium, was quoted at \$200 to \$225 per ton f.o.b. Niagara Falls, N.Y.

## USES OF TITANIUM AND ITS COMPOUNDS

Titanium, as such, does not appear ever to have been put to industrial use, but alloyed with other elements, and in chemical combination, it is employed for a number of purposes, mostly in a very small way. Considering the comparative abundance of titaniferous ores and the many suggested applications of titanium compounds, commercial development has been slow, probably because most of the suggested applications call for purer titanium compounds than have heretofore been obtainable at prices low enough to make their use profitable.

The useful products into which titanium may enter as an important constituent may be classified as follows:

1. Metallic alloys: such as ferrotitanium and ferrocarbontitanium.

2. Incandescent media for lighting purposes: arc-lamp electrodes and filaments for incandescent lamps.

<sup>Bulletin of the Imperial Institute, vol. 15, 1917, p. 85.
Bulletin of the Imperial Institute, vol. 15, 1917, p. 84.
Carteret and Devaux, La titane et ses emplois industriels: Age de Fer, 25 Octobre</sup> 1920; cited by Coffignier in Chimie et Industrie, Avril 1922, p. 661.

4 Min. Res. of the U.S., for 1918, pp. 809-810; for 1919, p. 719.

# 3. Colouring materials:

(a) Pigments, in the paint industry.

(b) Dyes and mordants, in the textile and leather industries.

(c) Refractory colouring material in the ceramic industries.

## 4. Miscellaneous.

At the moment of writing, by far the most extensive use of titanium is in the form of ferroalloys for the purification of steel. The amount consumed in other forms, for all other purposes combined, is only a fraction of the amount used in this way. The comparatively insignificant amount of titanium ore required to fill the present demands even of the iron and steel industry, as compared with the total supplies of titanium ores available, becomes evident when it is stated that less than two pounds of titanium are necessary for the production of a ton of the treated steel, and that less than 12,500 tons of 20 per cent titanium ore, such as ilmenite, would provide all the titanium required in the production of the 2,500,000 tons of titanium treated steel that, it is estimated, were manufactured in the United States in 1918. Recent developments in the use of titanium white as a substitute for white lead and zinc white would indicate, however, that the paint industry will probably be the most important consumer of titanium compounds in the future.

## 1. METALLIC ALLOYS

A considerable number of alloys of titanium with one or more other elements have been prepared and tested for various purposes, but the only ones that have actually appeared on the market as articles of commerce are the iron-titanium alloys (ferrotitanium and ferrocarbontitanium), cuprotitanium, and manganotitanium.

## TRON-THANHUM ALLOYS!,2

Possibly on account of the traditional superiority of the pig irons made from titaniferous ores, the alloys of titanium with iron were the first to receive attention, and they are now, by reason of their extensive use in the purification of steel and iron, commercially the most important of all the titanium compounds.

When titaniferous iron ores are smelted in the blast furnace a little of the titanium is reduced and alloys with the iron; most of it, however, goes into the slag, as TiO<sub>2</sub>, without reduction. But when titaniferous pig iron is converted into steel the titanium it contains is completely oxidized during the steel-making process and all passes into the slag as titanium dioxide (TiO<sub>2</sub>). Consequently, whether or not titanium improves the qualities of the pig irons containing it (a question on which metallurgists are not all in agreement) the quality of steel cannot be improved by the presence of titanium in the pig iron from which it is made. To affect its quality, the titanium must be added to the finished steel, and as there are practical difficulties both in the production

46410-7

<sup>1</sup> Anderson, R. J., The metallurgy of titanium; Jour. Franklin Inst., vol. 184, 1917, pp. 495-500, 503-508 and 637-650.

<sup>2</sup> The distribution and uses of litanium ores; Bull. Imp. Inst., No. 1, 1917, pp. 82-98. 3 Harbord and Hall, The Metallurgy of Steel, London, 1911, pp. 402-403. 4 Raymond, R. W., Trans. Amer. Inst. Min. Engrs., vol. 21, 1892, p. 278.

of metallic titanium and in its incorporation in molten steel, it is added in the form of an iron-titanium alloy.

The iron-titanium alloys were first produced by reducing highly titaniferous iron ores under suitable conditions in the blast furnace, but it was found impossible to obtain alloys high in titanium in this way, and they are now manufactured either in the electric furnace or by the aluminothermic method. There are at the present time two distinct kinds of ferrotitanium alloys on the market; one, called ferrocarbontitanium, containing 15 to 18 per cent titanium, about 6 per cent carbon, and the balance iron, is made in the electric furnace and sold, in 1920, at \$200 to \$250 per ton; the other, a carbon-free product, is manufactured by the thermit process and sells at a higher price. Both are used for the same purposes.

## Ferrocarbontitanium

The form in which titanium is chiefly used in the steel industry in America is the alloy known as ferrocarbontitanium, manufactured by the Titanium Alloy Manufacturing Company, of Niagara Falls, N.Y. The present position of this material in the commercial world is due almost entirely to the efforts of A. J. Rossi, who has devoted more than 30 years to the problems involved in its production and use. He and the company founded on the results of his labour, have been, very appropriately, styled "the alpha and omega of the titanium industry in America." As it comes on the market this material, which is used as a final deoxidizer and denitrogenizer in the treatment of steel, contains from 5 to 9 per cent of carbon, practically all in the form of graphite, and is guaranteed to contain at least 15 per cent titanium. A typical analysis is as follows:—

## Analysis of Ferrocarbontitanium

Silicon	1.41
Titanium	15.79
Carbon	7.46
Manganese	0.11
Aluminum	0.80
Phosphorus	0.05
Sulphur	0.08
Iron (by difference)	74.30
Aluminum Phosphorus Sulphur Iron (by difference)	0.05

The first commercial iron-titanium alloy made by Rossi in the electric furnace was a practically carbon-free product. The method employed in its production was to first melt aluminum in ingots or other convenient form in the electric furnace, then add pig iron or iron scrap to the charge, and, finally, rutile. The temperature was then raised to the point at which a reaction commences whereby the aluminum combines with the oxygen of the rutile and sets titanium free. The latter at once alloys with the molten iron, and in this way alloys containing 10 to 75 per cent titanium with only 0.12 to 0.75 per cent carbon can be produced in quantity. Ilmenite, or titanic iron ore can be used in place of rutile, in which case it may not be necessary to add pig iron or scrap. The aluminum first reduces the iron oxides in the ore, and in the iron thus formed, the titanium dissolves, as it is in turn reduced, yielding carbon-free ferrotitanium. The production of ferrotitanium from ilmenite in this way, however, is a more costly process than that in which rutile and

scrap are used, since a great deal of aluminum is required to reduce the iron

oxides, and iron can be more economically reduced in other ways.1

In its present practice, the Titanium Alloy Manufacturing Company has discarded the aluminum bath and substituted a method whereby oxides of iron and titanium are reduced with carbon in the electric furnace. At one of their plants, titaniferous iron ore or slag or rutile is mixed with the proper amount of carbon (coke) and charged into a Siemans electric furnace on top of a molten bath of scrap iron or steel. Reduction of the ore takes place in the arc and the resulting alloy is tapped off. The ore used has the following composition: TiO<sub>2</sub>, 34·36 per cent; FeO, 50·53 per cent; SiO<sub>2</sub>, 4·14 per cent; Al<sub>2</sub>O<sub>3</sub>, 2·20 per cent. If the slag produced is high in titanium, it is re-charged to the furnace. The first alloy obtained contains 10 to 15 per cent titanium, 5 to 9 per cent carbon, and 0.35 per cent to 1.00 per cent silicon; it can be refined, using rutile as a decarbonizing agent, to a final product carrying less than one per cent carbon.<sup>2</sup> To reduce the amount of titanium lost through oxidation, Rossi, in a patent issued in 1912,3 calls for the introduction of lime (CaO) into the bath of molten iron, to be followed by mixed titanium oxide, lime, and carbon, the reaction in this case being given as follows:—

# $TiO_2 + CaO + 5 C = Ti + CaC_2 + 3 CO.$

Rossi also devised and patented a method of concentration to produce an alloy rich in titanium from low grade ores.4 The ore containing 15 per cent titanium dioxide, 80 per cent oxide of iron, and 5 per cent of gangue, was smelted in an electric furnace with just sufficient carbon to reduce the iron but leave all the titanic acid unreduced in the slag. By this means a good quality of pig iron was obtained, and all the titanium was concentrated in the slag. The latter was then used for the production of ferrotitanium in the ordinary way. Analyses of ferrotitanium alloys so made, are:-

	TO 00 FF 04
Titanium	53.92 75.84
I I Camum.	0.291 $0.311$
Carbon	0 40 2
	0.059 0.042
Dhognhorits	0 000

### Ferrotitanium

By ferrotimium is meant the carbon-free binary alloy now produced by the well known (inkladianide aluminothermic process. It is very similar to the alloy produced by our of Rossi's earlier electric furnace processes already alluded to, both depending for their success on the strong reducing power of aluminum. Most ferrotitanium of German origin is made by the Goldschmidt process, and, in America, it is the praces employed by the Metal and Thermit Corporation (Goldschmidt Thermit Communy) of New York, the only company besides the Titanium Alloy Company engaged in the manufacture of ferrotitanium alloys on this continent.

Rossi, A. J., On the manufacture of ferroalloys in general and of ferrotitanium in Prossi, A. J., On the macular are of ferroalloys in general and of ferrotitanium in particular in the electric furnace: Electrochem. Ind., vol. 1, 1903, pp. 523-526.

2 Anderson, R. J., The metallurgy of dianium: Jour. Franklin Inst., vol. 184, 1917, 2 Anderson, 2 Anderson, 3 L., The metallurgy of dianium: Jour. Franklin Inst., vol. 184, 1917, 2 Anderson, R. J., The metallurgy of dianium: Jour. Franklin Inst., vol. 184, 1917, 2 Anderson, R. J., 1908, 466, 1912, 3 U.S. Patent No. 1,019,528, Mar. 5, 1912, 3 U.S. Patent No. 668,266, Feb. 19, 1901, 4 Electrochem. Ind., vol. 1, 1903, p. 526; also U.S. Patent No. 668,266, Feb. 19, 1901.

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In the Goldschmidt process the metallic oxides to be reduced are mixed with aluminum in the proper proportions, all the ingredients being in a finely powdered condition. By means of a suitable fuse, the mixture is then ignited and the temperature raised at some point in it to that at which aluminum and oxygen combine. Once started a rapid reaction takes place; titanic acid (TiO2) is reduced to the metal, and the aluminum combining with the liberated oxygen forms alumina, according to the following equation.1

# $3 \text{ TiO}_2 + 4 \text{ Al} = 3 \text{ Ti} + 2 \text{ Al}_2 \text{O}_3$

These alloys contain 5 to 6 per cent of aluminum, which is said to be advantageous inasmuch as it facilitates the incorporation of the alloy in molten iron and steel. An analysis of such an alloy follows:-

# Analysis of Carbon-free Ferrotitanium

Silicon	1 to 1.5 per cent
fitallithin	25 " "
Calpun.	nil
Manganese	nil
Aluminum	3 (0 0
Phosphorus	0·05 " " " 0·01
Sulphur	67.44 to 68.94 per cent
Iron (by difference)	01.44 to 09.24 ber cent

The Goldschmidt Company also put up so-called "titanium thermit" by means of which the liquid titanium alloy is formed in the bath of molten metal to be purified. It consists of an intimate mixture of powdered aluminum with oxides of iron and titanium, in suitable proportions, confined in a sheet iron receptacle. This, fastened to the end of an iron rod, is stirred into the molten metal, the temperature of which is sufficient to start the reaction between the metallic oxides and the aluminum. This method of use is said to be specially suitable for adding small quantities of titanium to cast iron.2

The Goldschmidt products have been used for a number of years in many European steel works, as a final deoxidizer during casting. They are also used to some extent in America, where, however, the less expensive electric furnace product, ferrocarbontitanium, is the favourite.

Ferrotitanium versus Ferrocarbontitanium.—Metallurgists are practically all agreed that a small amount of titanium in some form can be used to advantage for the final purification of practically every grade of steel-high carbon, low carbon, and special alloy steels; but as is natural when two different commodities with similar functions are put on the market by rival manufacturers, there has been some controversy concerning the relative merits of ferrocarbontitanium and the carbon-free product for this purpose. It has been stated<sup>3</sup> that the titanium in ferrocarbontitanium is present in the form of titanium carbide, in microscopic particles held in a matrix similar to grey cast iron, while in the carbon-free product it is alloyed with iron as the simple metal. The

<sup>1</sup> Goldschmidt, Hans, Aluminothermics: Electrochem. Ind., vol. 1, 1903, pp. 527-534. The production of pure metals free from carbon by the aluminothermic method: Electrochem. Ind., vol. 2, 1904, pp. 145-147.

<sup>2</sup> Goldschmidt, Hans, Titanium thermit: Electrochem. and Met. Ind., vol. 3, 1905,

Goldschmidt, Hans (Discussion), Trans. Amer. Electrochem. Soc., vol. 20, 1911, p. 271. 8 Comstock, G. F., Titanium and its effects on steel: Jour. Soc. Chem. Ind., vol. 34, 1915, p. 55.

dispute seems to hinge primarily on the matter of alleged carbides in the electric furnace product. These, it has been claimed, prevent the ready solution of the alloy in steel and, hence, render it unable to do the same work as the carbon-free alloy. There is also the question as to the effect on the steel of the aluminum always present in the carbonless alloy.

Dr. Hans Goldschmidt, the advocate of carbon-free ferrotitanium, says¹ that carbon-free metals dissolve more readily than do the same metals when they contain carbon, because in the latter the carbon is present in the form of carbide. He states further<sup>2</sup> that the aluminum in carbonless ferrotitanium is a useful ingredient having a threefold purpose; first, in that it causes the titanium to alloy better with the molten steel; second, that it facilitates the removal of the waste products formed during the process of purification—since the titanic acid formed by the titanium is, by itself, very infusible, as is also the alumina formed by the aluminum, but when both metals are added at the same time titanate of aluminum, a much more fusible compound, is formed, that by reason of its fluidity readily frees itself and rises to the surface of the molten bath; third, in that the aluminum acts on the steel before the titanium and thus relieves the latter of the rough preliminary work of deoxidation that can be more advantageously done by aluminum.

Rossi<sup>3</sup> and Slocum<sup>4</sup> strenuously combat the view that the carbon in ferrocarbontitanium is in the form of carbide and that the presence of carbon impairs the usefulness of the alloy. In support of their contention, Slocum cites a number of analyses that show the carbon to be present almost entirely as graphite and only an inconsiderable proportion in the combined form, and claims there is no ground for the belief that the carbon present in the triple alloy is there mainly as carbide, that, on the contrary, titanium appears to act very much like silicon in that it causes a separation of the carbon as graphite.

# Analyses showing Condition of the Carbon in Ferrocarbontitanium

$\begin{array}{cccccccccccccccccccccccccccccccccccc$		Graphite	Combined carbon
2	1	9.601	0.147
0.118		9.179	0.120
		7.012	0.130
		6.234	0.118

Slocum<sup>3</sup> further states that his experience is that the carbon-free alloy is not adapted for use in the iron and steel industry, for which purpose an alloy containing 5 to 8 per acre purbon is demanded, and that his attempts to use a carbon-free iron-titanium alloy containing 5 to 10 per cent aluminum resulted in almost complete failure, as the aluminum made the steel brittle and its oxides remained as inclusions in the metal.

The density of an allor to be used as the ferrotitanium alloys are used has an important bearing on the facility with which it can be incorporated in the

<sup>1</sup> The melting point and its relation to alloying capacity: Met. and Chem. Eng., vol. 9,

Rossi, A. J., The manufacture of titanium and its alloys: The Mineral Industry, vol. 9, 1900, p. 716. 4 Slocum, C. V., Titanium in iron and steel: Trans. Amer. Electrochem. Soc., vol. 20, 1911, pp. 265-268.

5 Loc. cit.

molten steel. In this respect ferrotitanium made by the aluminothermic process and ferrocarbontitanium are on an approximately equal footing, since both have densities of about 6.20 to 6.40.

Whatever the relative merits of the two types of iron-titanium alloys, both are in actual use and for the same purposes. So far as America is concerned, ferrocarbontitanium appears to be in more general demand<sup>1</sup>, while aluminothermic ferrotitanium commands the higher price.

Titanium in the metallurgy of steel.—The presence of minute quantities of certain elements in finished steel produces seriously deleterious effects on the physical properties of that metal, so that it is a matter of great commercial importance that such injurious impurities be eliminated as completely as possible during the process of manufacture. Among the worst of these impurities are oxygen and nitrogen,2 which are always present, and for the removal of which titanium is specially adapted by its great affinity for both. Titanium will remove traces of oxygen that do not yield to ordinary deoxidizers with their concomitant carburizing action, and it is one of the few elements that will readily unite with the inert gas, nitrogen. Moreover, the titanium dioxide formed by its reaction with oxygen combines with the slag occluded in the steel, and, by lowering its melting point, imparts to it a fluidity that greatly facilitates its separation from the metal.3 It is for these reasons that titanium is a valuable final deoxidizer, or scavenger, for the purification of steel. For this purpose it may be used either alone or alloyed with iron, but, aside from the difficulties involved in the production of pure metallic titanium, certain practical considerations preclude the use of the pure metal. Its high melting point (1850° C.) and a specific gravity less than that of iron would cause it to float on molten steel, so that it would be practically impossible to bring about its uniform diffusion throughout the bath. In practice, therefore, it is always used in the form of a ferroalloy; steel works in the United States usually specifying one containing 10 to 15 per cent titanium, since one of that composition readily dissolves in molten iron and steel.

In steel works practice it is customary to add the ferrotitanium alloy in the casting ladle after the other deoxidizers, such as ferromanganese and ferrosilicon, if they also are to be used. The alloy, broken into small-pieces, is shovelled, cold, into the molten steel as it is being tapped into the ladle, when the latter is about one-third full. It is advisable that as much as possible of the alloy be rabbled into the molten steel before the slag begins to run, otherwise much of it will be wasted in the vigorous reaction that takes place between titanium and the molten silicates, and because it is difficult for the alloy to make its way down through the slag to the metal beneath. After the titanium alloy has been thoroughly incorporated in the bath, the ladle should be allowed to stand for a few minutes before pouring, in order that the titanium may have time to react with the impurities and to give the entrained slag and oxides an opportunity to separate from the steel and rise to the surface. There is no danger of the bath chilling by thus holding it, since the reaction brought about

<sup>&</sup>lt;sup>1</sup> Anderson, R. J., Make progress in ferro-alloys: Iron Trade Review, vol. 66, 1920, p. 1122.

<sup>&</sup>lt;sup>2</sup> Chatelier, Henri le, Congress of Metallurgists, Belgium, 1905.

Braune, ——, Stahl und Eisen, Jahrgang 26, 1906, pp. 1357, 1431 and 1496. Tholander, Dr., Stahl und Eisen, Jahrgang 29, 1909, p. 1594. Stoughton, B., U.S. Pat. Office, Proc. Ser. No. 463,610.

by the titanium is exothermic: the addition of the iron-titanium alloy as followed almost immediately by a rise in the temperature of the fused steel due to the heat given off by the reaction of the titanium with oxygen and nitrogen. Stable oxides and nitrides of titanium are formed, which rise to the surface and are removed as slag. The amount of titanium alloy to be added varies with the grade of steel treated and with the results desired. In general, less than one per cent of the weight of the steel, equivalent to less than two pounds of metallic titanium per ton of steel poured, is all that is necessary. Unless more has been added than is required to unite with all the impurities, all the titanium will pass off in the slag. If, however, an excess of titanium has been added some of the surplus will remain alloyed in the steel, where it does no harm and may even improve the quality.

The effect of adding titanium is to break up dissolved oxides and nitrides and remove occluded gases, thus rendering the finished steel denser and closer grained and free from blowholes and pinholes. Piping in the ingot is also reduced and the segregation of sulphur, phosphorus, and carbon diminished.1

Titanium-treated steel castings are free of brittleness, heat less under the tool, and may therefore be machined more rapidly than other steels of like carbon content. It makes possible the removal of a greater amount of slag, and, by preventing segregation, produces a more homogeneous metal.

The first really extensive use to which the titanium-iron alloys were put after their introduction into the steel industry was the purification of bessemer rail steel, and in 1910 over a quarter of a million tons of that steel was so treated; but, with the substitution of open hearth for bessemer steel rails, the use of titanium alloys fell off rapidly, as it was not thought that the already superior qualities of the open hearth metal were susceptible of sufficient improvement by a titanium treatment to warrant the extra expense. Further experience, however, has indicated that not only open hearth but practically all steels can be benefited by treatment with ferrotitanium alloys,2 so that although the output of titanium-treated rail steel is now but a small fraction of what it was formerly, the total amount of titanium-treated steel produced has been growing steadily and has now reached very considerable proportions. The following figures from various annual volumes of The Mineral Industry will serve to illustrate this:-

<sup>1</sup> Von Maltitz, E., Der Einfluss des Titans auf Stahl besonders auf Schienenstahl: Stahl und Eisen, Jahrgang, 29, 1909, pp. 1593-1602.

Waterhouse, G. B.. The influence of thankm on segregation in bessemer rail steel: Proc. Amer. Soc. Testing Materials, vol. 16, 1910, pp. 201-211.

2 Venator, Wilhelm, Ueber Verwendung von Titan-Legierungen in der Stahl-Industrie; Stahl und Eisen, Jahrgang 30, 1910, p. 651.

Janssen, W. A., The use of titanium in the manufacture of steel castings, Amer Foundrymen's Assoc., Cleveland meeting, 1916.

### Annual Production of Titanium-Treated Steel in the United States

		production1
09	256,759	1,000,000 2,000,000 2,200,000 2,200,000 2,400,000

<sup>&</sup>lt;sup>1</sup>Estimates based on sales of ferrotitanium alloys and information furnished by manufacturers of ferrotitanium.

In addition to their present comparatively insignificant use as a superpurifier of rail steel, the ferrotitanium alloys are now being applied in the treatment of steel and iron rolls, where resistance to abrasion and shock is desirable; for steel castings, in foundry practice; for both low and high carbon steel chain; for gears, pinions, tires, die plates and heads; propellor shafts, driving rods, and other forgings; for tool (crucible) and automobile steels; for cast iron ingot moulds; for acid pots, and chilled iron car wheels.

During the war, experiments in connection with the conservation of manganese showed that ferrotitanium could, in part, be successfully substituted for ferromanganese for some purposes. One steel plant was able to reduce the usual amount of ferromanganese fifty per cent by the addition of ferrotitanium in the ladle.<sup>1</sup>

Aside from its properties as a scavenger, interest has also been manifest in the effect of titanium as an actual alloying element in steel. Used in this way it is said to improve the qualities of nickel, chromium, and manganese steels. Manganese steel containing 11 per cent manganese, 1 per cent carbon, and up to 0.40 per cent titanium is readily forged and rolled, and can be machined with comparative ease<sup>2</sup>, and experiments made in the Westinghouse research laboratory have shown that the addition of two per cent of titanium to ironnickel alloys renders them capable of being forged at ordinary forging temperatures.<sup>3</sup> It is reported that certain noteworthy developments have been made in the case of complex titanium-bearing steels for armour plate and ordnance, but detailed data are not available.<sup>4</sup>

The effect of titanium on cast iron.—There are fewer data available concerning the effects produced by treating cast iron with titanium than is the case with steel, and the results recorded are somewhat contradictory, probably

 $<sup>^{\</sup>rm 1}$  Anderson, R. J., Make progress in ferro-alloys: Iron Trade Review, vol. 66, 1920, p. 1122.

Anderson, R. J., Jour. Franklin Inst., vol. 184, 1917, p. 506.
 Bull. Can. Min. Inst., No 99, July, 1920, p. 543.
 Mineral Industry, vol. 29, 1920, p. 678.

because the elements with which titanium reacts were present in different

proportions in the various cast irons treated.

One thing that seems to stand out clearly is that the addition of ferrotitanium reduces the chilling property in cast iron, probably by removing sulphur, nitrogen, and oxygen, all of which tend to increase the chill and for all of which titanium has a strong affinity. The removal of these elements may also serve to explain the discordant effects observed on the strength, etc., of the iron, for, though titanium helps to eliminate the detrimental sulphur, it also removes the highly beneficial oxygen, so the net result will depend on the relative proportions in which these two elements were present in the untreated metal. It may be here incidentally remarked that the presence of oxygen in cast iron, once firmly believed to be highly detrimental, just as it is still in the case of steel, has been shown by recent research to be, on the contrary, highly beneficial.<sup>1</sup>

The reduction in chilling power brought about by the use of ferrotitanium alloys in cast iron requires the use of more manganese for its restoration, which is highly objectionable, and it seems very doubtful whether any benefit will be found by prolonged experience to result from the use of titanium in cast iron, in spite of the fact that its action on the matrix of the iron itself may be to strengthen it. Its influence in removing oxygen in good castings is extremely detrimental, and this is probably not offset by any other beneficial action which it may have; on the other hand, where particularly good castings are not required, there is no call for the use of any treating material whatever.<sup>1</sup>

Titanium contained in pig iron to be used for making steel can have no effect on the steel made therefrom, as it is all oxidized and removed as slag

in the process of steel making.

If it is desired to employ titanium as a deoxidizer of east iron, the usual method of procedure in foundry practice is to scatter a predetermined amount of ferrocarbontitanium or ferrotitanium, as the case may be, along the runner prior to tapping, so that when the cupola is tapped the titanium alloy will be washed into the ladle with the stream of iron. As in steel practice, the ladle should be held for some minutes to permit complete chemical reaction to take place, and slag should not be allowed to come into contact with the alloy, for it attacks ferrotitanium with avidity and thus destroys much of its usefulness. For most purposes two to four pounds of a 15 per cent titanium alloy per ton of iron is requisite and sufficient.<sup>2</sup>

# Cuprotitanium

Copper when molten very readily absorbs gases and becomes oxidized. Upon cooling, the occluded gases are largely given off, producing at times ebullitions sufficiently violent to interfere with proper casting and leaving the metal full of blow holes and other imperfections. For this reason copper cannot be cast in sand direct from crucibles, in sand moulds made in flasks, or in such moulds as are used for casting iron. By the addition of small

McGraw-Hill Book Co., 1918, pp. 480-490; see pp. 469-481 for effect of oxygen on cast iron, and pp. 481-484 for objections to too much manganese.

2 Anderson, R. J., Jour. Franklin Inst., vol. 184, 1917, p. 886.

amounts of titanium in the form of a copper alloy solid castings free from blow holes are said to be ensured.1 Aluminum bronze treated with titanium, while considerably lighter than phosphor or manganese bronze, is said to be equal to it in strength and to be little affected by sea-water.

An alloy of copper and titanium containing from 5 to 12 per cent of the latter element is said to be valuable for addition to copper castings.2 The alloy is manufactured in the electric furnace by the Rossi process, which calls for the reduction of rutile in a bath of aluminum to which copper has been added for the purpose of alloying with the reduced titanium. Otherwise the process is identical with that for the production of ferrotitanium, but ilmenite cannot be used, as the product must contain no iron.

An alloy containing 93 per cent copper, 5 per cent titanium, and 2 percent magnesium is said to be still more efficient for degasifying copper and its alloys.

The method of using cuprotitanium is practically the same as that employed when ferrotitanium is added to steel. Little has been published relative to its ise.

#### Ferrosilicotitanium

When it is desired to add both silicon and titanium during the refining of steel, the use of an alloy containing titanium, silicon, iron, and carbon has been advocated as preferable to the addition of the silicon and titanium separately. Such an alloy has been prepared in the electric furnace by reducing rutile and "silver sand" with carbon; the iron may be added either as scrap or as iron oxide together with sufficient additional carbon for its reduction. An analysis of ferrosilicotitanium produced in this way is:-

#### Analysis of Ferrosilicotitanium

Iron	 	(	43.69
			33.70
Silicon	 		14.23
Carbon	 		$8 \cdot 32$
Aluminum	 		0.08
Calcium	 		Trace

It is proposed that the alloy be added to the molten steel as it runs from the furnace, in exactly the same way that ferrotitanium is used. Although it was patented in 1909, this alloy has not apparently been put to any extensive industrial use.

# Ferrocuprotitanium

By substituting copper for silica in the production of ferrosilicotitanium, an alloy similar to the latter in other respects may be obtained for subsequent incorporation in copper.4

<sup>&</sup>lt;sup>1</sup> Electrochem. and Met. Indust., vol. 7, 1909, p. 88.
<sup>2</sup> Min. and Sci. Press, vol. 99, 1909, p. 355.
<sup>3</sup> Electrochem. and Met. Indust., vol. 7, 1909, p. 538. Jour. Soc. Chem. Indust., vol. 29, 1910, p. 636.
<sup>4</sup> Jour. Soc. Chem. Indust., vol. 29, 1910, p. 636.

# Manganotitanium

An alloy of manganese and titanium, made by the thermit process, was brought out by the Goldschmidt Thermit Company for use as a deoxidizer of bronze. It contains 30 to 35 per cent titanium.1

# 2. INCANDESCENT MEDIA

Titanium in the form of its natural compounds, ilmenite and rutile, as well as in the artificial compound, titanium carbide, has been employed to a noteworthy extent as one of the ingredients of electrodes for arc lamps; and filaments of metallic titanium have been tested experimentally in incandescent electric lights. Experiments with titanium dioxide as a material for Welsbach gas mantles indicated that it was not suitable for that purpose. The value of titanium and its compounds for illuminating purposes is said to lie chiefly in their high fusion and vaporization points and great radiation efficiency; also, the spectrum furnished by titanium is one of the richest given by any of the elements.

# Arc Light Electrodes

As early as 1878, Thomas A. Edison took out an English patent covering the use of titanium dioxide (rutile) in arc lamps, but the first United States patent mentioning the use of any compound of titanium in the manufacture of are lamp electrodes was one issued in 1890, which called for the addition of some difficultly fusible substance, such as titanic iron (ilmenite), to the carbon of the electrodes in order to improve the steadiness and intensity of the light. Later, a series of experiments were carried out in the research laboratory of the General Electric Company, in order to ascertain what available material would give the maximum light efficiency when used as arc electrodes. The work covered a large number of elements, compounds, and mixtures, but none of the other substances tried gave as high efficiency in candle power per watt as material which contained the element titanium.3

Probably the most extensively used electrode containing titanium is that employed in the so-called magnetite lamp, in which the cathode consists of a mixture of magnetite with 15 to 20 per cent rutile and some chromite. The magnetite gives conductivity to the electrodes when cold, as the other oxides are conductors only when hot, the rutile ensures a high luminous efficiency, and the chromite retards the consumption and increases the life of the electrode. This lamp which first found favour in the United States is now also used in Europe to some extent. One disadvantage connected with it is that it cannot be used in an alternating current circuit.4

In January, 1907, a patent (U.S. Pat. No. 840,634) was granted for an arc light pencil consisting in whole or in part of an alloy of titanium with some metal possessing greater electrical conductivity. Such electrodes have been made directly from ferrotitanium or from a mixture of magnetite and

<sup>1</sup> Anderson, R. J., Jour. Franklin Inst., vol. 184, 1917, p. 501. 2 Hess, F. L., Mineral Res. of the U.S., 1907, pt. 7, p. 721. 3 Weedon, W. S., The titanium arc: Trans. Amer. Electrochem. Soc., vol. 16, 1909,

<sup>&</sup>lt;sup>4</sup> Bull. of the Imp. Inst., vol. 15, 1917, pp. 95-96. Barrows, W. E., Electrical Illuminating Engineering.

rutile, the oxides in the latter case being subsequently partly reduced. Those made in the second way are said to give the most satisfactory results. Among the advantages claimed for this electrode are: greater durability, low amperage required for its operation, and a light approaching sunlight in character.<sup>1</sup>

More recently, patents have been granted covering the use of titanium carbide (TiC), either alone or mixed with other substances, in the manufacture of electrodes, and much effort has been spent in an endeavour to perfect this type of light. The composition of the electrodes of this class varies chiefly in the proportion of titanium carbide to the carbon usually mixed with it in the course of manufacture. One English patent (No. 18,220 of 1912) calls for a mixture of 96 per cent titanium carbide, 3 per cent copper oxide, and 1 per cent lithium fluoride. In one type of carbide electrode the titanium carbide is surrounded by an iron tube, and in another the outside is plated with copper, these devices being used to increase the conductivity and prevent oxidation while the lamp is burning.

The titanium carbide used in the manufacture of electrodes should be free from iron or silicates, since these tend to lower its melting point. It is made from rutile, in the electric furnace, by mixing that mineral with an excess of ground coke and heating to the highest temperature available, the result being the reduction of the titanium dioxide and the simultaneous formation of titanium carbide. The mode of procedure is to fill the furnace with a mixture of rutile and coke and then pass a heavy electric current through the centre of the mass. The carbide is formed only in the central part of the furnace near the electrodes, but the unreduced material surrounding it serves to prevent access of air and the formation of nitrides. The product is a silver-white porous mass that tends to crystallize in small cubes with hollow sides.

The characteristics of the titanium-carbide light are those of the so-called luminous arc, that is, an extremely luminous inner path, with very little light from the outer mantle and none from the electrodes themselves; in the carbon lamp nearly all the light radiates from the incandescent electrodes. Titanium-carbide electrodes are said to be much more durable than carbon electrodes and to be almost free from the unpleasant hissing and decrepitation characteristic of the latter. They operate most satisfactorily on a constant current system.<sup>2</sup>

Titanium sub-oxide, also, has been used in the manufacture of arc lamp electrodes. The sub-oxide is made by mixing rutile with carbon in the ratio of 7 to 1 and heating in the electric furnace. As the melting point of the sub-oxide is considerably lower than that of the carbide, its preparation is relatively easy. As is also the case with the titanium carbide arc, some good conductor, like copper or carbon, is used for the anode. The arc obtained is almost identical in character with that of the carbide lamp, and the light distribution is the same. It is, however, steadier in operation and has greater lighting efficiency. The great objections to the sub-oxide electrodes are their rapid consumption, the formation of troublesome deposits on the anode, and the fact that they cannot be operated on an alternating current system. Both the carbide and sub-oxide electrodes require a special starting device, as a non-conducting

<sup>&</sup>lt;sup>1</sup> Ladoff, Isador, The titanium arc: Jour. Ind. and Eng. Chem., vol. 1, 1909, pp. 711-723. See also pp. 642-644.

Watson, T. L., and Taber, S., Vir. Geol. Sur., Bull. No. III-A, 1913, p. 279.

Weedon, W. S., the titanium arc: Amer. Electrochem. Soc. vol. 16, 1909, pp. 217-224.

layer of titanium dioxide forms at their tips and causes difficulty in starting again after the lamps have been once in operation and have been allowed to cool.1

A number of other mixtures containing some compound of titanium have been proposed for use as luminous arc electrodes. For example, U.S. Patent No. 1,112,458 of 1914 calls for one consisting largely of carbon, with some calcium titanate, an organic salt of titanium, and a titanium halogen compound or alkali titanofluoride; the use of cerium titanofluoride is covered by English Patent No. 13,988 of 1912. Such a mixture may consist of 45 per cent carbon, 35 per cent cerium titanofluoride, 10 per cent potassium fluoride, and 10 per cent barium fluoride. The light is steadied by the addition of potassium or sodium fluoride, and the barium fluoride is added to correct the colour of the flame. A little silicate, borate, carbonate, tungstate, or molybdate of sodium may also be added to the mixture to minimize the etching effect of the fluorides on the glass globes. Titanic oxide mixed with calcium cyanamide, cryolite, and carbon is suggested in English Patent No. 11,792 of 1912, and fused oxides of titanium, tungsten, and rare earths, in molecular proportions, in U.S. Patent No. 1,161,173 of 1913.

# Incandescent Lamp Filaments

Metallic titanium has been tried for the manufacture of filaments for incandescent lamps, the processes of manufacture and use being covered by many patents. The intense white light emitted is said to be due to selective radiation rather than to the preponderance of certain colour lines in their spectrum. They must be used in an atmosphere of neutral gas or in a vacuum to prevent exidation of the metal.

A trial lot of about 1,000 incandescent lamps furnished with titanium filaments was produced in the United States in 1906,2 but certain technical difficulties connected with their manufacture have so far prevented their coming

into general use.

One method of overcoming the difficulty involved in preparing pure metallic titanium and subsequently drawing it into wire is to force a colloidal solution of titanium hydroxide through a small nozzle, and, after drying the fine thread thus produced, to reduce it to the metallic state in hydrogen. Should there be the slightest trace of carbon present in the filament, such as may be derived from the vaporized oil from the pump during the exhaustion of the bulbs, the efficiency of the lamp will be so impaired as to render it practically useless.3

For business reasons, the manufacturers who are endeavouring to produce a commercially successful incundescent titanium lamp are, naturally, reticent concerning the progress made, so that little has been made public concerning their

manufacture.

<sup>1</sup> Weedon, W. S. Loc. cit.
2 Hess, F. L., Min. Res. of the U.S., 1906, p. 530.
3 The Electrician, vol. 58, 1907, p. 892.
Mining World, vol. 33, 1910, p. 230.
Bull. Imp. Inst., vol. 15, 1917, p. 96.

#### Incandescent Gas Mantles

Although oxide of titanium has been tried for that purpose, it does not seem to be suitable material for the manufacture of incandescent gas mantles.<sup>1</sup>

#### 3. COLOURING MATERIALS

# (a) Pigments in the Paint Industry

One of the first uses proposed for any compound of titanium was that made many years ago—that ferrocyanide of titanium be substituted for the poisonous arsenical Schweinfurth green then so widely used. The suitability of various titanium compounds for pigments has been suggested many times since; but until recently none of them have actually been manufactured and put on the market for that purpose.

A protective coating for iron and steel is said to be formed by mixing a powdered titanium ore with asphalt, and, if the product is to be used as a paint, adding turpentine. Such coatings can also be formed by covering the metal to be treated with a layer of the titanium mineral mixed with carbonaceous material, and heating in a furnace for several hours.<sup>2</sup>

Barnes says titanium tannate can be used effectively as a pigment in watercolour paintings, the colour—depending on the conditions of preparation of the pigment—varying from brick red to almost pure yellow.3

If a titaniferous iron ore such as ilmenite is roasted at a temperature below incipient fusion and then crushed under water, it is capable of yielding a finely divided product of a yellow or red colour suitable for paint pigments.4

Light yellow pigments suitable for use in rust-preventing paints can be made, according to English Patent No. 10,368 of 1911, by digesting titaniferous iron ore with sulphuric acid and then roasting the mass at a temperature high enough to decompose the sulphates.

Titanium white.—The first titanium compound to be used in the paint industry in important quantity, however, is the recently developed white pigment, titanium white, which is now being put on the market in competition with white lead and zinc white. It is produced commercially in Norway by the Titan Company at Fredrickstad, and in America, under the trade name of titanox, by the Titanium Pigment Company, Inc., at Niagara Falls, N.Y. It seems likely to become in the near future the most important commercial product of titanium.5

A brief outline of the Norwegian company's process follows<sup>6</sup>: The raw material on which the manufacture is based is ilmenite, carrying 25 to 45 per cent titanium dioxide (TiO2), from the well known ilmenite deposits at Blaafjeld and Storgang, near Soggendal, on the southwest coast of Norway. The mineral is sorted, crushed, classified, and washed over tables at the mines, then transported by rail and boat to Fredrickstad for further treatment. At Fredrickstad, after being dried and subjected to fine grinding, it is thoroughly

<sup>Jour. Franklin Inst., vol. 186, p. 612.
Jour. Soc. Chem. Indust., vol. 12, 1893, p. 1046.
Jour. Soc. Chem. Indust., vol. 15, p. 420.
Jour. Soc. Chem. Indust., vol. 29, 1910, p. 1023.
The Mineral Industry, 1920, vol. 29, p. 677.
The Chem. Trade Jour. and Chem. Engr., Sept. 25, 1920, p. 407.</sup> 

mixed with sulphuric acid in a special mixing apparatus, and then transferred to the decomposition plant, in which a reaction between the sulphuric acid and the ilmenite is brought about by gas heating. The resulting sulphates of iron and titanium, mixed with sulphuric acid and undecomposed mineral, are delivered by the decomposition apparatus continuously in the form of cakes of convenient size, which are crushed, ground, and leached with water, the iron and titanium sulphates going into solution. The fine particles of undecomposed material that remain suspended in the solution must now be separated from it, and considerable practical difficulty was encountered before this could be successfully accomplished. Filtration in any form proved useless, and at the present time large settling tanks, provided with devices to hasten the speed of settling, are employed. The ferric sulphate in the perfectly clear solution from the settling tanks is next reduced to the ferrous state by electrolysis; and, by boiling the reduced solution in a vessel provided with internal steam coils, the titanium is precipitated as dioxide, in the form of a white amorphous powder. The removal of ferrous sulphate and free sulphuric acid from the titanium dioxide precipitate is effected by means of a washing process, the extreme fineness of the precipitate and the corrosive action of the acid solution rendering any method of separation by filtering practically impossible. The last traces of sulphuric acid are removed by the addition of barium carbonate, so the final product contains a certain amount of barium sulphate. How the last of the iron is removed is not divulged—as little as 0.02 per cent is sufficient to discolour the product—but it is suggested that it is rendered harmless by the addition of substances that form colourless compounds with iron. It may be for this purpose that 15 per cent of phosphoric acid in the form of calcium phosphate is added immediately after the barium carbonate.1

The washed and neutralized titanium dioxide is next calcined. Ground to such fineness that 99.95 per cent will pass through a mesh of 5,000 to the square centimetre and mixed with linseed oil and a little zinc white, after thorough incorporation in an edge-runner mill, it is ready for use. An analysis of a sample of one of the first Norwegian titanium whites put on the market

yielded2:-

Loss on ignition	1.34
Titanic acid	66.64
Titanic acid	12.48
Barium sulphate	0.37
Silica	10.54
Phosphoric acid	20 0 2
Lime	8.06
Oxide of iron	0.72

The plant at Fredrickstad is capable of handling 8,000 tons of ilmenite

per annum, equivalent to about 3,000 tons of dry titanium pigment.

The advantages claimed for titanium white as compared with white lead and zinc oxide are: that it possesses greater covering power than either, is not poisonous like white lead, is chemically inert, and, unlike zinc oxide, exerts no saponifying action on linseed oil.

 $\Lambda$  microscopical investigation of the covering power of pigments has led to the conclusion that covering capacity depends, among other things, on the numerical difference between the refractive index of the pigment and that of

<sup>1</sup> Chimie et Industrie, vol. 7, No. 4, April, 1922, p. 653.

<sup>2</sup> Loc. cit., p. 654.

the binder used. In this connection, it is interesting to note that if the refractive index of linseed oil be taken as unity then that of zinc oxide is 1.34, of white lead 1.36, of amorphous titanic acid 1.48, and of crystalline titanic acid 1.80. The final calcination in the manufacture of titanium white is for the purpose of taking advantage of this fact and increasing the covering power of the precipitated titanium dioxide by changing it from the amorphous to the crystalline form. This, according to patents issued to the Titan Company, in July, 1918, may be brought about by heating the amorphous precipitate to a suitable temperature either alone or in the presence of a catalyser, such as a halogen compound.

The properties of a white titanium pigment of American origin, titanox, are briefly described in a booklet published by the Titanium Pigment Company, of Niagara Falls, N.Y.<sup>2</sup> The details of the process by which it is manufactured, however, have not been made public. The following information con-

cerning titanox has been culled from the company's booklet:-

The material from which titanox is at present manufactured is obtained by concentrating a beach sand containing ilmenite, rutile, zircon, and monazite, found at Mineral City in Florida. The different valuable minerals are separated by appropriate means and the ilmenite and rutile used for the manufacture of titanium pigments, alloys, and other products, the zircon for refractory purposes and the manufacture of zirconium oxide, and the monazite is worked up for cerium and thorium to be used in the manufacture of gas mantles.

According to their behaviour when mixed with such a vehicle as linseed oil, white pigments may be divided into two classes: opaque white, or light reflecting, pigments characterized by their property of imparting a white colour and opacity to the paints of which they are components, and the so-called inert, or extender, pigments, such as whiting, barytes, gypsum, china-clay, silica, and asbestine, which, though white in the dry or powdered state, have little or no opacity or hiding power when mixed with oil and spread as paint. Heretofore, the trade has had to rely entirely on white lead, zinc oxide, and lithopone for material of the first class, but the commercial development of the white titanium pigments has made available a new material, titanox, having greater stability and inertness as well as greater covering power than any of these.

The titanox pigments are of two types: those consisting essentially of pure titanium dioxide, and certain composite pigments in which the titanic acid is precipitated upon or with a base, or extender, pigment. Though processes have been developed for the manufacture of the first type, and the products have given excellent results when used in various paint compositions, the present cost of manufacturing pure titanium dioxide is such as to reduce its general commercial application, so that the composite pigments of the second type are of greater importance from a commercial viewpoint.

In the manufacture of the composite titanium pigments, titanium dioxide is precipitated from solution upon and into coalescence with a base, or extender, pigment, which is either precipitated simultaneously or, having been previously formed, is held suspended in the solution during the precipitation of the titanic oxide. The mixed precipitate is then filtered off, washed, dried, calcined, and milled. The composite titanium pigment of American

<sup>&</sup>lt;sup>1</sup> Abstracts, American Chemical Soc., vol. 13, 1919, pt. 1, p. 383; and pt. 2, p. 1938.

<sup>2</sup> Titanox Pigments, Titanium Pigment Company, Inc., Niagara Falls, N.Y., and 94
Fulton St., New York City, 1921.

origin that has been most fully investigated is composed of titanic oxide on a precipitated barium sulphate base and is known as titanox "B". It is this material that is referred to as titanox, throughout the remainder of this report.

Titanox "B" contains titanium dioxide, 25 per cent, and barium sulphate, 75 per cent. Its specific gravity is 4.30, and its bulking figure, i.e., its weight in pounds per solid (U.S.) gallon, is 35.8. It is said to possess greater covering power than any other commercial white pigment, to be unaffected by the action of light or gases, and to be particularly inert towards other pigments and with all vehicles and liquids, so that it may be used with all kinds of gums and resins without danger of livering, hardening, or granulation.

Titanox, it is claimed, can be used to advantage in both interior and exterior work. It is peculiarly adapted for use in a wide variety of modern industrial enamels and undercoatings for application by spraying, brushing, and dipping, and for air drying and baking. Alone or combined with extender or other pigments it is suitable for interior flat wall and gloss paints, and for enamel paints, both for interior use and of the spar-varnish type for exterior use.

In linseed oil paints for exterior use, it should be mixed with zinc oxide, for though they do not check or crack, paints made with titanox alone dry to an elastic film that is too soft to resist the accumulation of dust. The addition of zinc oxide corrects this condition. Paints composed of titanox alone in a linseed oil vehicle also tend to chalk, but when zinc oxide is added in proper proportions they are exceptionally durable, substantially free from chalking, and retain their original brilliance of colour to a remarkable degree.

Titanox has also given excellent results, it is stated, when used as a pigment in the printing ink, wall paper, rubber, oilcloth, and linoleum industries.

Commercial titanium white pigments of Norwegian origin are, like titanox, composite pigments. They come on the market in three grades: extra containing about 65 per cent titanic acid, standard, about 25 per cent, and special with about 15 per cent. According to Coffiguier experience with them in France confirms all the claims that have been made for their remarkably useful properties. He gives the relative weights of the different grades of Norwegian titanium white, zinc white, and white lead respectively, required to hide equal areas of black surface, as follows:—

	55
Extra titanium white	64
Standard titanium white	95
Special titanium white	0 10
Zinc white	100
White lead	160
White lead	

The chief obstacle to their more extensive use in France he attributes to their high price as compared with other white pigments, which is due in part to adverse exchange and customs duties. He goes on to point out, however, that if the superior covering power of the titanium whites is taken into consideration their use is just as economical as, if not more so than, that of the other white pigments, even at current prices. The higher price of the titanium whites remains, nevertheless, a distinct commercial drawback.

<sup>1</sup> Coffignier, Ch., Fabrication et propriétés au blanc de titane: Chimie et Industrie, Vol. 7, No. 4, 1922, pp. 651-661.

<sup>46410-8</sup> 

Coffignier also outlines what are said to be more economical methods for the production of titanium white than any now in use: methods that have been devised by French chemists with a view to lowering the cost of production both by simplifying the process of manufacture and by the simultaneous production of salable by-products. Patents are pending for these processes. They have not yet been put into practice.

Carteret-Devaux process.—In the process of Carteret and Devaux, titanium ore mixed with carbon and a flux is reduced in a furnace at red heat, and dry chlorine gas is passed into the mixture. Under these conditions different chlorides are formed successively according as the temperature rises; silicon chloride, for example, forms at 59°, and titanium chloride at 135°. The reaction thus affords the basis for an easy method of separating these two chlorides from each other and from ferric chloride by a simple fractional distillation.

The solution of titanium chloride obtained may be precipitated either with barium carbonate or by hydrolysis. The inventors favour the latter method, with the recovery of hydrochloric acid. The resulting titanic acid is free from iron. A special feature of the method is the facility with which a clean separation can be made when using complex ores and the different metals or their chlorides recovered for use in other industries.

As may be seen in the accompanying diagrammatic representation of the process, the by-products that may be recovered are: hydrochloric acid, either for sale or for regeneration of the chlorine, coloured pigments from the wash waters, and electrolytic iron.

Paquet-Andreu process.—The originality of Paquet's and Andreu's process consists, as does that of Carteret and Devaux, in uniting in an appropriate sequence a number of previously known reactions so as to bring about the simultaneous production of iron-free titanic acid and certain valuable byproducts. The process is carried out in three stages:—

(1) Carbonitride of titanium and sodium cyanide are produced in a revolving furnace by heating titanium ore mixed with carbon and sodium carbonate in a current of nitrogen, according to the following equation:—

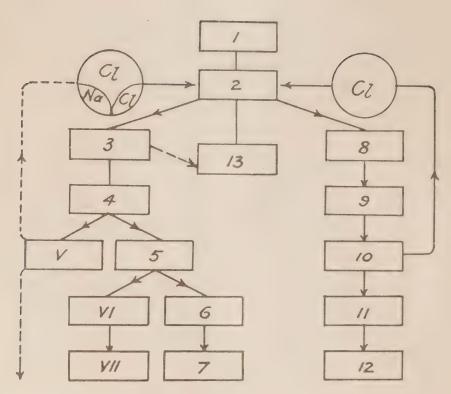
$$10 \text{ TiO}_2 + 10 \text{ Na}_2\text{CO}_3 + 62 \text{ C} + 28 \text{ N} = \text{Ti}_{10}\text{C}_2\text{N}_8 + 20 \text{ Na CN} + 50 \text{ CO}.$$

A number of patents have already been issued covering the preparation of sodium cyanide from sodium carbonate, carbon, and nitrogen by heating them together in a furnace, but these previous processes have all failed commercially, largely because the high temperature necessary to bring about the reaction quickly destroyed the apparatus used. In the presence of titanium ore, however, it is claimed that the reaction can be brought about at a temperature below 1000°.

(2) The mixture is next subjected to the action of steam; titanate of soda, ammonia, and hydrocyanic acid being formed, as follows:—

$$Ti_{10}C_2N_8 + 20 Na CN + 80 H_2O =$$
  
10  $Na_2TiO_3 \cdot 4 H_2O + 16 NH_3 + 12 HCN + 10 CO + 20 H.$ 

The disengaged gases are absorbed, the ammonia in sulphuric acid, the hydrogen cyanide in a soda solution.



- Concentration of the ore
- Reduction and chlorination Distillation of TiCl<sub>4</sub> Hydrolysis of TiCl<sub>4</sub>
- Precipitation of pure TiO2 5.
- Recovery of HCl Washing of TiO<sub>2</sub> V.
- 6.
- Wash waters VI.
  - Preparation of neutral white
- Treatment of wash waters for coloured pigments Solution of the Fe<sub>2</sub> Cl<sub>6</sub>
  Reduction to Cl<sub>2</sub>
  Electrolysis yielding iron and chlorine
  Treatment of the electrolytic iron VII.
  - 8.
  - 9.
- 10.
- 11.
- Pure merchantable iron 12.
- Undissolved material and residues from lixiviation, for special treatment 13.
- Production and desiccation of the chlorine C1.

Fig. 5. Diagram of the Carteret-Devaux process.

(3) The mass remaining after the treatment with steam is exhausted with hot water and the resulting solution of titanate of soda is decomposed with a current of carbonic acid, thus:—

$$Na_2TiO_3 \cdot 4 H_2O + CO_2 = Ti(OH)_4 + Na_2CO_3 + 2 H_2O.$$

By the Paquet-Andreu process there is thus obtained, besides titanic acid, ammonium sulphate and sodium cyanide as by-products, and the sodium carbonate is regenerated. Theoretically, 735 kg. of sodium cyanide will be produced for each metric ton of titanic acid.

The method appears to be an economical one. The ammonium sulphate is a valuable fertilizer and sodium cyanide is used in the treatment of many gold ores.

# (b) Dyes and Mordants in the Textile and Leather Industries

As early as 1885 attention was called to the fact that titanium compounds could be used in mordanting yarns, and in 1896 it was suggested that they might be used for dyeing leather; but it was, at first, found difficult to produce titanium salts sufficiently free from iron for these purposes. Later, TiO (SO<sub>4</sub>Na)<sub>2</sub>·2H<sub>2</sub>O sufficiently free from iron to be used in mordanting wool was put on the market and, afterwards, the double oxalate of potassium and titanium, which was specially adapted for dyeing leather. In 1901 the use of titanous salts, such as titanous sulphate, was proposed as stripping agents.<sup>1</sup>

At the present time titanium compounds are successfully used, to a small extent, both in Europe and America, as mordants and dyes for cotton, wool, and other textiles, and they may also be used to colour and mordant paper and paper pulp. The oxalates and double oxalates of titanium and an alkali metal, particularly, are used as mordants and are said to produce with tannin a yellow colour of great durability. Titanous chloride also has been used as a mordant, and titanous sulphate as a mordant and stripper,<sup>2</sup> while double pyrophosphates of titanium and an alkali metal are said to be applicable to textile and other substances without danger of injury to the fabric.<sup>3</sup>

Titanium potassium oxalate, TiO(C<sub>2</sub>O<sub>4</sub>K)<sub>2</sub>·2H<sub>2</sub>O, which is produced commercially in a high state of purity, is probably more extensively used as a mordant and dye in the textile industries than any of the other titanium salts, though the corresponding sodium compound is cheaper and is said to make a satisfactory substitute.

Titanous chloride, TiCl3, and titanous sulphate, Ti2(SO4)3 are also manufactured in a commercial way, both in America and in Europe, for use in the textile colouring industries. They are said to be the most powerful soluble acid-reducing agents known and to be more efficient substitutes for stannous chloride, the best acid-reducing agent hitherto employed in dyeing and printing textiles. In textile printing titanous salts may be used either for discharge or resist work.

<sup>&</sup>lt;sup>1</sup> Barnes, Joseph, Properties and uses of some titanium compounds: Jour. Soc. Dyers and Colourists, vol. 35, 1919, pp. 59-62. Abstracted in Abstracts Amer. Chem. Soc., vol. 13, 1919, pt. 2, p. 2454.

Uses of Titanium; Mining World, vol. 33, 1910, p. 230.
 Jour. Soc. Chem. Ind., vol. 15, 1896, p. 420; and vol. 18, 1899, p. 15
 London Mining Journal., July 13, 1907, p. 65.

These salts are also used as stripping, or bleaching agents. In dyeing textiles such as wool, cotton, and silk, it sometimes happens that a wrong shade is produced or that the dyeing takes place unevenly. When this occurs it is necessary to either remove or reduce the colour, i.e., strip, and re-dye or dye a darker shade. Ordinary bleaching agents cannot be used for stripping with most colours without danger of injury to the fabric, but if such dyed goods are passed through a hot dilute solution of suitable titanous salts the colour is destroyed without injury to the cloth. Titanous salts in very dilute solution are also used to clear up white portions of fancy coloured goods. The double sulphate of titanium and sodium is, commercially, the most important titanium salt used as a stripper.

In the leather industry, potassium titanio-oxalate, TiO(C<sub>2</sub>O<sub>4</sub>K)<sub>2</sub>·2H<sub>2</sub>O, is used on a rather extensive scale, both in America and in Europe, for staining and dyeing leather, particularly chrome-tanned leathers.2 It is said to produce on the fibre of the leather a yellowish-brown titanium tannate which is extremely fast to light and the action of alkalis and hence is especially suitable for shoe leathers. It is extremely soluble in hot water and can be applied to tanned leather either in the dye bath or by brushing. Used as a striker (precipitant) for logwood on leathers, it produces an intense permanent black, and is said not to make the leather brittle and less durable as do iron salts when employed for this purpose.3

Titanium oxalate, and the double tartrates and lactates of titanium and the alkali metals, are also used in leather dyeing. Before the war titanium lactates were advertised under the trade name corichrome, as mordants and strikers for use in the leather industry. The preparation of these compounds is described in English Pats. Nos. 22,629 of 1901, 23,188 of 1901, 14,921 of 1902, and 27,597 of 1902. A full account of their uses has been published in the Leather Trades

Review.4

# (c) Refractory Pigments in the Ceramic Industries

There is a small but steady demand for specially prepared grades of rutile, such as those sold under the trade name titanellow, for use in the ceramic industries.5

These are used chiefly for colouring artificial teeth, of which over 8,000,000 are said to be produced annually, in the United States, and in all of which rutile is the colouring matter employed. By the addition of one-half to four or five per cent of rutile to the paste of kaolin, silica, and feldspar from which they are moulded, a series of standard coloured teeth are produced, and, from these, teeth of any shade desired for matching with the adjoining natural teeth can be selected.

On account of their fire-resisting properties, titanium dioxide, some compounds of uranium, and oxide of iron are the only three substances practically available for the production of the colour yellow in the body of porcelain ware or in underglaze painting on porcelain. For this purpose, rutile is cheaper than compounds of the rare element uranium and more satisfactory than oxide

<sup>1</sup> Barnes, Joseph, Jour. Soc. Dyers and Colourists, vol. 35, 1919, pp. 59-62.

<sup>&</sup>lt;sup>2</sup> Leather World, vol. 8, 1916, p. 15. <sup>3</sup> Titanium Mordants: Shoe and Leather Reporter, 1908, pp. 19-20. <sup>4</sup> Leather Trades Review, vol. 46, 1913, pp. 183 and 266. <sup>5</sup> Titanium: The Mineral Industry for 1919, vol. 28, p. 685.

of iron, but great care is necessary in its use in order to obtain the result desired. Alone, it imparts to porcelain a fine yellow colour, the exact shade of which depends on the amount of rutile present, and it may be used in conjunction with other substances to produce various secondary colours, but unless extra precautions are taken to shield the porcelain while in the furnace from the action of reducing gases, the rutile (TiO2) may be reduced to Ti2O3, which imparts a deep copper-red colour to the ware. Sometimes also a blue colour is inadvertently produced, probably through the formation of titanium nitrides and the carbonitride by the action of CO and atmospheric nitrogen on the rutile at high temperatures. In the manufacture of artificial teeth, where rutile is the pigment universally employed, the walls of the muffle in which the teeth are baked are carefully coated with clay before each firing in order to prevent the ingress of furnace gases. In view of the extra care and precautions necessary in the burning when rutile is used for colouring porcelain wares, it would appear that its use for this purpose will probably be commercially successful only where the results sought are such as to justify considerable added expense in the preparation of the finished goods.<sup>1</sup>

Titanium dioxide also finds a small but regular use as a crystallizer, for producing crystalline glazes on art pottery. Practically the only other crystallizers available are ZnO and MnO.<sup>2</sup>

Titanic acid when added in certain proportions develops opacity in enamels, the shade produced being chiefly greyish to yellowish-white. The TiO<sub>2</sub> used is prepared by freeing rutile from iron and other impurities.<sup>3</sup>

A series of tests carried out by Messrs. Landrum and Frost<sup>4</sup> led to the conclusion that there is a most promising field in developing the commercial use of titanium enamels. Their resistance to chemical action is much greater than in the case of ordinary enamels, their high gloss offers great resistance to abrasion, and they have a high heat resistance. Though there are several technical difficulties connected with their use that have still to be overcome, their good qualities outweigh their bad ones, and it should be possible to develop titanium enamels having a marked practical value.

#### 4. MISCELLANEOUS USES

Certain titanous salts are said to be finding an extended use on account of their great power as acid reducing agents. Sodium titanous sulphate, Ti(SO<sub>4</sub>)<sub>2</sub> ·Na<sub>2</sub>SO<sub>4</sub>·3H<sub>2</sub>O, and titanous chloride, TiCl<sub>3</sub>, are employed as reducing agents for various purposes; the former can be thus used in the preparation of other titanous salts. Titanous sulphate, said to be the strongest acid reducing agent now known, is used to decolourize old paper, dyed fabrics, etc.<sup>5</sup> It has also been recommended for detecting copper in dyed goods. Titanous sulphate, Ti(SO<sub>4</sub>)<sub>2</sub>, may also be employed in analytical work to detect the presence of

<sup>&</sup>lt;sup>1</sup> Snelling, W. O., Titanium; Min. Res. of the U.S. for 1901, pp. 271-278.

<sup>&</sup>lt;sup>2</sup> Purdy, R. C., and Krehbiel, J. J., Crystalline glazes: Trans. Amer. Cer. Soc., vol. 9, 1907, pp. 325 et seq.

<sup>&</sup>lt;sup>3</sup> Grünwald, Julius, and Hodgson, H. H., Raw Materials of the Enamel Industry, J. B. Lippincott & Co., Phila., 1914, p. 126.

<sup>&</sup>lt;sup>4</sup> Landrum, R. D., and Frost, L. J., Titanium enamels: Jour. Amer. Cer. Soc., vol. 3, 1920, pp. 316-321; abstracted in Raw Materials, vol. 4, No. 2, Feb., 1921.

<sup>5</sup> Zeitsch. Phys. Chem., vol. 62, 1908, p. 147.

fluorine.1 Titanous chloride is used in laundries, for removing iron stains and for clearing coloured goods that have run in the wash.2

Titanium dioxide, TiO2, is said to form a very efficient catalyst in the esterification of acetic acid.

The logwood titanium compound makes a permanent ink, deep black in colour, which undergoes no change after using.

On account of the extreme brilliance with which it burns, titanium has

been used in pyrotechny.3

Titanium nitride has been suggested as a basis for fertilizers and other nitrogeneous compounds, and patents have been issued covering the manufacture of titanium nitride for this purpose as a by-product of the smelting of titaniferous iron ores.4

Moissan<sup>5</sup> found that Ti<sub>2</sub>N<sub>2</sub> has a hardness greater than that of the ruby, and sufficient to slowly polish diamonds.

During the later years of the great war considerable titanium tetrachloride, TiCl4, was used for the production of smoke screens to conceal the movements of vessels and troops.6

A little rutile has been used in the manufacture of glass,7 and some natural crystals of rutile are sufficiently clear to be valuable as semi-precious stones for gem purposes.8

<sup>1</sup> Hess, F. L., Min. Res. of the U.S. for 1906, p. 530.

2 Bull. Imp. Institute, vol. 15, 1917, p. 97.

3 Cahan, Ed., and Wootton, W. O., The mineralogy of the rarer metals. Griffin & Co.,

London, 1912, p. 111.

4 Watson, T. L., and Taber, Stephen, Contary of the titanium and apatite deposits of

Wirginia: Bull. 3-A, Virginia Geol. Sur., 1913, p. 287.

Virginia: Bull. 3-A, Virginia Geol. Sur., 1913, p. 287.

5 Moissan, H., Ann. Chim. Phys., vol. 7, p. 229.

6 Hess, F. L., Titanium: Min. Res. of the W.S. for 1918, pp. 809-810.

6 Hess, F. L., Titanium: Min. Res. of the W.S. for 1918, pp. 823-331.

war: Trans. Amer. Electrochem. Soc., vol. 35, 1919, pp. 323-331.

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<sup>8</sup> Kunz, G. F., Gems and precious stones, New York, 1890, pp. 193 et al. 8 Kunz., G. F., Gems and precious stones, New York, 1890, pp. 193 et al.



# INDEX

	Page
A	
Alberta, titaniferous magnetite	88
Algoma district, rutile	45
Alma island, titaniferous magnetite	60
American Rutile Co94, 9	5, 96
American Titanic Ore Co	49
Analysis—	
Beauce co. magnetite	9, 70
Bedford tp. magnetite	75
Blithfield tp. magnetite	76
Bloc range magnetite	70
Bouchard mine ilmenite	48
Bristol tp. ore	71
Burmis titaniferous magnetite	88
Canadian titaniferous iron ores	35
Cap Rond titaniferous magnetite	66
Chaffey mine magnetite	74
Clarendon tp. ore	71
Clarke City deposits	65
Coulomb mine ilmenite	9, 50
Eagle Lake mine titaniferous magnetite	75
ferrosilicotitanium	106
ferrotitanium alloys	99
Furnace mine ilmenite	52
gabbro from Rivière des Rapides	
Gagnon gabbro	64
General Electric Co.'s mine ilmenite	51
Glen prospect ilmenite	48
Horton tp. titaniferous magnetite	76
Hull tp. ore	71
ilmenite	24
Ivry mine ilmenite	54
Kenogami tp. magnetite	60
Lake St. John co. titaniferous magnetite	60
Litchfield tp. ore	71 65
Marguerite river ore	65
" titaniferous magnetite	73
Matthews mine magnetite	80
Minden tp. magnetur	77
Orton mine magnetite	61
Parry Sound district magnetite8	
pig iron made at St. Urbain	27
Pine lake ore	79
Quebec Seminary ilmenite	53
Ricketts property ore	78
rutile-bearing ilmenite, St. Urbain	43
St. Charles ore	58
Sanford concentrates	29
Sudbury district magnetite	82
manufactor to hometite	0, 71
The man and magnetite	68
The state of the court of the c	66
magnetita Saine hay iron ratio	5, 86
t	5, 66
. at 1 day and an another the transfer of the	
Arc light electrous from transfer.	93

В

			ь						P	AGE
									1	
Bachman, acco	unt of smeltin	g experiments			• • • • • •		• •	• •	11	29
Bad Vermilion									<del>1</del> Ι,	
Basanomelan							m 4	• • -1	<b>⊢</b> ~	23
Bay of Seven I	Islands, titanii	ferous magneti	te				7, 4	Ι, :	51,	60
Beauce co., ilm										44
		netite								69
Bedford tp., tita	_									74
Beresford tp., i										53
		agnetite								68
Berzelius, expe										13
Bibliography								• •		36
Bignell ilmenite							• •	• •		52
Blessington mil										n -
Blithfield tp., m										75
Bloc range, ma									4 57	70
Bouchard's mir									′	48
Bourget tp., tit								• • •	οι,	58
Bowron, conclu								• •		28
Bristol tp., tita	iniierous magi	ietite	• • • •				• •	• •		71
Brome co., tita	nic acia, etc					• • • • •	• •	• •		44
Buckingham tp									n.4	44
Buckman and									94,	95
Burmis, Alta.,								• •		41
	titaniierous m	agnetite					• •	• •		88
			C							
Canada, produc										93
Canadian Titar									50,	
Cap Rond, tita:										66
Carteret-Devay									1	.14
Ceylon, ilmenit										96
Chaffey iron m										73
Chaloupe river										66
Charlevoix co.,										46
Chicoutimi co.,										58
Chrichtonite								• •		23
Chromium in t								• •		24
Clarendon tp.,								• •		71
Clark City dep										65 24
Cobalt in titan									9 1	
Confignier, facts Concentration									ο, 1	87
"	experiments tr		"	Degrosbois						69
66	6.6	66	66	Grondin ore						67
6.6	6.6	66	66	Rivière des						63
6.6	66	66	66	St. Charles						59
Coulomb's min	e St Urbain			ot. Charles	010	* * * *		• •	47	48
Cran de Fer fa	alls titaniform	is magnetite						• •	¥ 6 9	62
Creusot Steel V	Works source	of vanadium								33
Cuprotitanium.	Works, source	or vanaurum,.	• • • •					• •	1	.05
J D D A O VA OWALL CALLE		• • • • • • • • •					• •	• •	_	00
			775							
			D							
Décharge rang	e. prospecting	on								53
Degrosbois dep	osits.									68
Dicksbergite	44.44.									21
Dulieux, ilmeni	ite denosit at s	St. Urhain des	cribed					• •		42
Dunlop, D. A.,	Nipissing dist	rict property	21000							81
2,		- La paropored .					• •			01
			E							
			100							
Eagle Lake mi	ne									74
Edison, Thos.	A., patent, tita	nium in arc la	amps.						1	.07
Electric smelti	ng of titanifer	ous iron ores.								31
										J

# F

Fakenthal, B. F., smelting of titanium ores  Ferrocarbontitanium	Pagi
Ferrocuprotitanium. Ferrosilicotitanium. Ferrotitanium.	. 106 . 106
Flesche d'amour.  Forbes, David, smelting experiments with titaniferous ores.  Fredrickstad, production of titanium white at.  Frontenac co., deposits.	. 100 . 45 . 27, 28 . 110
Fuel consumption smelting titaniferous magnetites excessive	27, 30
° G	
Gagnon deposit.  General Electric Co., rutile deposit operated by.  " " workings, St. Urbain.  Glamorgan tp., ore of varied character.  Glen prospect, ilmenite.  Goldschmidt, Dr. Hans.  " production of titanium alloys.  " Thermit Co.  Goodwin, result of experiments as to vanadium.  " Dr. W. L., diamond drilling, Rainy River district.  Grattan tp., magnetite.  Gregor, Rev. Wm., discoverer of titanium.  Grondin blast furnace.  " mine.	. 41, 43 . 47, 50 . 78 . 47 . 101 . 14 . 99 . 34 . 84 . 76 . 13
н	
Haliburton co., deposit of varied character. Halifax co., rutile. Hamilton Blast Furnace Co. Hastings co., deposits.  " rutile. Haystack mountain, titaniferous magnetite. Hematite, association of with ilmenite.  " Templeton tp. Heskett, J. A., experiment smelting New Zealand titaniferous sand. Hinchinbrooke tp., titaniferous magnetite. Horton tp., titaniferous magnetite. Hull co., occurrences small.  " tp., titaniferous magnetite. Hunt, T. Sterry, mention of Bay of Seven Islands deposits.  " " " Beauce co., titaniferous magnetite. " " " rutile near St. Urbain noted by. Hunter, Mr., diamond drilling done for.  " " work of.	45 75 76 45 82 23 70 30 74 76 70
Ilmenite	19 20
" described" " first shipments from Canada" " found in two localities in Quebec" " how formed" " iron from at St. Urbain" " minor occurrences" " mixture of with magnetite"	25 93 41 24 27 56 32 45
" occurrences raw material for titanium white	110

	T3
	Pagi
Ilmenite (titanic iron) character of	22
" value of	96
Ilmenorutile	23, 45
Introductory	7
Iron-titanium alloys	97
Irons from titaniferous ores better than other irons	26
Ivry, Que., ilmenite, commercial importance	25
" " deposit	1, 95
" " operations	53
*	
J	
The second of the property of	31
Johnson, J. E., comments on use of titaniferous ores	ĐΙ
K	
Kemp, J. F., origin, etc., titaniferous magnetites	57
Kenogami tp., titaniferous magnetite	59
Kibdelophane	23
Kings co., N.S., quartz with rutile	45
Klaproth (German chemist) discovery of titanium	13
Kragerö, rutile deposits	22
L	
Lake St. John co., titaniferous magnetite	60
Lake tp., titaniferous magnetite	76
Lampadius, experiments by	13
Landrum and Frost, tests of titanium enamels	118
Leeds co., magnetite mine.	72
Leverin, H. A., determinations as to vanadium content	34
Litchfield tp., titaniferous magnetite	
Loughborough Mining Co., rutile deposit worked by	42
Lount tp., magnetite	80
Fount tp., magnetite	00
M	
McGregor, Rev. Wm. See Gregor.	
McIntyre Iron Co., owners deposit of titaniferous magnetite	29
McNaught tp., iron prespect	81
McVittie locations	81
Madagascar, ilmenite deposit	96
Madoc tp., rutile	45
Magnetic concentration	
Manganotitanium	
Marguerite river, magnetite deposits	107
Manguette tiver, magnetite deposits	65
Marmora tp., rutile	45
Maryland Steel Works, first extended application of titanium	15
Matthews iron mine	72
Menachanite	13
Menachite	13
Metal and Thermit Corporation	96
Minden tp., magnetite	80
Mineral City, Florida, beach sand carrying ilmenite, etc	95
Minette ores, vanadium in	33
Moisie Iron Co., iron from titaniferous sand	27
Moissan, experiments by	13
Molson mine	62
Mountain lake, titaniferous magnetite	81
Mushet, Robert, patents for utilizing ores of titanium	14
N	
New Zealand, experiment smelting titaniferous sand	9.4
Nickel in titanium-iron segregations	30
Nickel in titanium-iron segregations	24
Nilson, method of preparing titanium	
Nilson, method of preparing titanium	13
The state of the s	81

PAG
Norfolk and Western railway, use of titanium by
O
Obalski, J., Bay of Seven Islands deposits mentioned. 80 Olary, S. Australia, deposit of ilmenite. 95 Orton mine. 36, 76 Ottertail brook, titaniferous magnetite. 81 Outarde Falls deposits. 64
r .
Paquet-Andreu process       114         Parks property, Grattan tp., magnetite       76         Parry Sound district, magnetite       80, 81         Perovskite       19, 20         Peterson, method of preparing titanium       13         Pine Lake deposit       78         " " misleading description of       79         Pontiac co., occurrences small       70         Pope, studies of analyses of magnetites       36         Port Henry, N.Y., test smelting titaniferous magnetite       29         Pusey deposit. See Pine Lake       78
Q
Quebec Mines and Metal Co.69"Seminary, ilmenite found on lands of.53Queensland, rutile shipments.94
R
Rainy River district, extensive deposits titaniferous magnetite.  Renfrew co., deposits Ricketts property, titaniferous magnetite.  Rivière des Rapides, titaniferous magnetite.  Rossi, A. J., account of smelting experiments.  """ carbon in ferrocarbontitanium.  """ production of titanium alloys.  Rutile.  """ crystals for gem purposes.  """ ceneral Electric Co.'s workings.  """ in manufacture of glass.  """ prices of.  """ sources of production.  """ use of in ceramic industries.  """ various deposits mentioned.  """ workable deposit at St. Urbain.
St. Charles ore, concentration tests

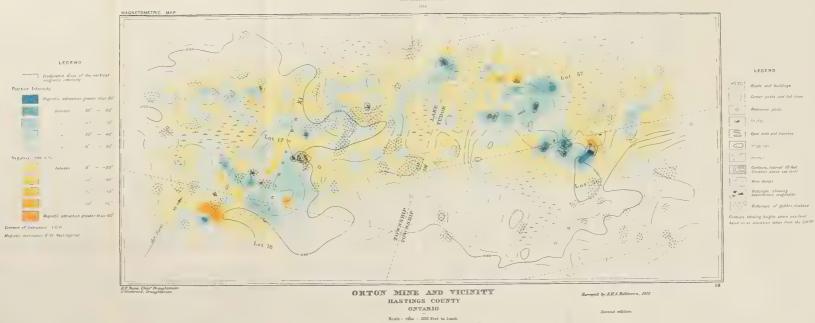
	PAGE
Saguenay county, titaniferous magnetite deposits	60
"river, titaniferous magnetite deposits	41
Sanford lake, N.Y., charcoal furnaces	27
Seine bay, titaniferous magnetite	
Shawinigan mine	66
"tp., titaniferous magnetite	66
Sheen tp., titaniferous magnetite	$\begin{array}{c} 72 \\ 101 \end{array}$
Slocum, carbon in ferrocarbontitanium	26
South Crosby tp., Chaffey iron mine	73
Strüverite	21
Sudbury district, iron prospect	81
Sutton tp., titanic acid, etc	44
Sweden, use of titaniferous ore	26
${f T}$	
Templeton tp., character of deposits	70
" rutile occurrence	44
Terrebonne co., ilmenite workings	53
" titaniferous magnetite	68
Thistle creek, Yukon, rutile	45
Thunder Bay district, titaniferous magnetite	82
"river, titaniferous magnetite	66
Titan Co., producers of titanium white	110
Titanellow	117
Titanic acid, how obtained	25
Titanic iron. See Ilmenite.	
Titaniferous magnetite	
" blast furnace smelting of	26
magnetic concentration of	32
more frequent than ilmenite in Canada	26
occurrences	56
origin and mode of occurrence	57 26
" ores objectionable in blast furnace	20
Titanium Alloy Manufacturing Co	
"annual production from Norway, the U.S. and Canada	93
" arc light electrodes	107
" attempts to reduce from its compounds	13
" colouring materials	110
" discovery of	13
" effect on cast steel	104
" incandescent media	107
" in the metallurgy of steel	102
" metallic alloys	97
minerals found in all classes of rock	19
prices of	96
never round native	19
" miscellaneous uses	118 18
" no satisfactory process for producing pure	18
" occurrences in Canada	41
" ores of	
" origin of name	13
" physical and chemical properties	15
" Pigment Co94, 11	
" preparation of metallic	
" sources, production and uses	93
" steel, annual production in U.S	104
" use of compounds of	14
" for dyes and mordants	116
" " lamp filaments	109
mantles	110
pigments in paint industry	110
in ceramic industries	117
uses of	96
" white	11, 110

	Pagi
Titanox  Tivani Steel Co  Travancore, India, ilmenite in monazite sands  Tudor tp., titaniferous magnetite	. 76
U	
Uddevalite	
V	
Vanadium content of Canadian titaniferous ores.  " in titaniferous magnetites.  " in titanium-iron segregations.  " sources of.  Venus' hair stone.  Versamm, F., patents for use of compounds of titanium.  Virginia, important producer.  " world's greatest producer of rutile.	24 . 34 . 45 . 14
w	
Wallace mine, Bay of Islands, rutile	. 42 52 . 13
Y	
Yankee mine. See Matthews mine. Yukon territory, rutile	. 45



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